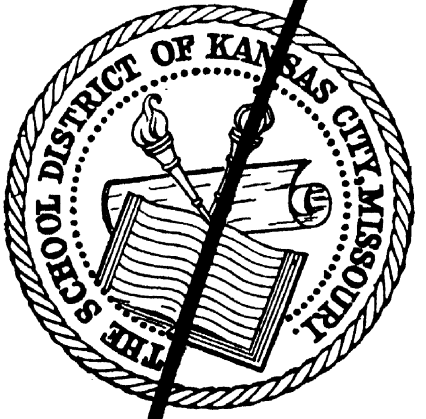
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Edited by

NATHANIEL HALL

and

G. B. HOGABOOM, Jr.

Associate Editors,
Metal Finishing

•

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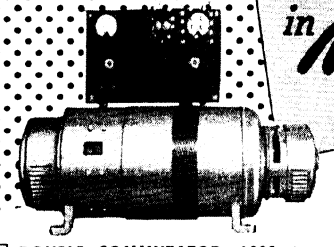
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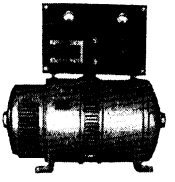
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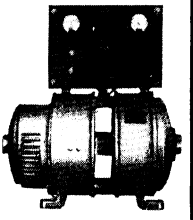
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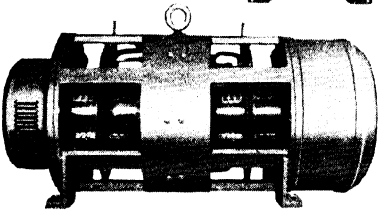
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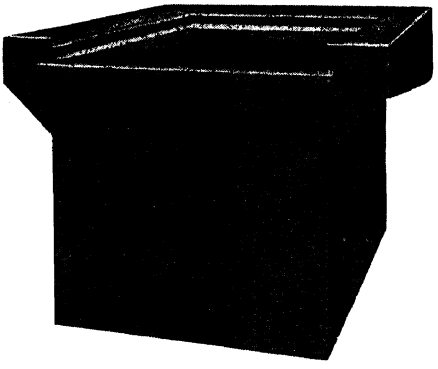
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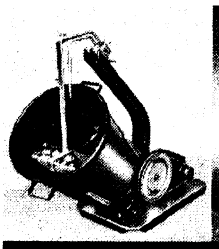
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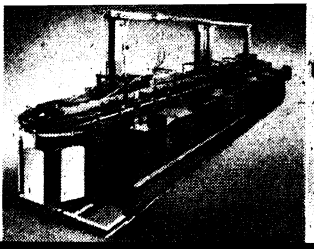
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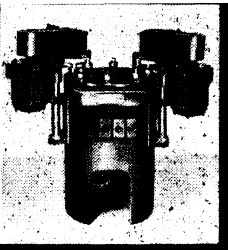
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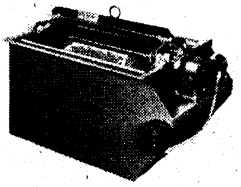
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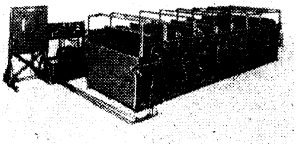
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PICKLING AND ACID DIPPING OF METALS

By Nathaniel Hall and G. B. Hogaboom, Jr.

ALUMINUM AND ITS ALLOYS

SHEET ALUMINUM

To produce a bright etched finish, sheet aluminum is first immersed in a hot alkaline etching solution such as 2-8 oz./gal. of caustic soda, soda ash or trisodium phosphate at 180-200° F. The period of immersion will depend on the degree of etching desired and if of sufficient duration will result in the formation of a smut on the surface. Since the etching solution is alkaline it is generally unnecessary to clean the metal first. However, if the work is oily, solvent degreasing or cleaning is advisable in order to prevent spotting.

The smut is removed by immersion for a few seconds in a nitric-sulfuric bright dip, such as is used for copper and copper alloys (see below), or in a mixture of 1 part nitric acid and 1 part water.

Aluminum sheet which has been heat treated often acquires a stain which is not removed by the alkaline etch. To remove this stain, the articles are immersed for one minute in:

Hydrofluoric acid	1 part
Nitric acid	1 "
Water	98 "

They are then rinsed and dipped in the above mentioned nitric or nitric-sulfuric acid bright dips.

Duralumin may be pickled in the following solution:

Sodium fluoride	1¼ oz./gal.
Sulfuric acid	13 "

This is followed by the acid bright dip.

CAST ALUMINUM

Aluminum castings, containing alloying metals such as silicon, iron, etc., acquire an adherent black or brownish film during the alkaline treatment due to dissolving of the aluminum out of the surface, the insoluble alloying metals remaining. A dip containing nitric and hydrofluoric acids is required for removal of this film, such as:

Nitric acid	3 gal.
Hydrofluoric acid	1 "

Room temperature. Ventilation required.

Immersion for 15-30 seconds is generally sufficient for aluminum die castings and 1-2 minutes for sand castings. Originally lead lined tanks were used, coated with a mixture of 1 part beeswax and 4 parts paraffin and placed in a water bath in order to prevent the wax from softening and to keep the rate of reaction from becoming excessive. However, the action is so rapid that the bath heats up suffi-

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ciently to soften the wax when used in production and results in wax films on the work after a short operating period. Organic lining materials are now available to withstand the action of the nitric-hydrofluoric acid mixture. Details regarding suppliers of such linings may be obtained from *Metal Finishing*.

COPPER AND ITS ALLOYS

PICKLING

1. Sulfuric Acid Pickle:

Sulfuric acid	1 gal.
Water	9 "

Temperature: 125-175°F.

On copper and copper alloys containing more than about 85% copper, oxide films will contain a high percentage of cuprous oxide (Cu_2O) which is not easily removed by sulfuric acid pickles, and the addition of an oxidizing agent is desirable. Examples of such solutions are:

2. Dichromate Pickle:

Sulfuric acid	1 pt./gal.
Sodium dichromate	2-4 oz./gal.

Temperature: Room to 175°F.

3. Ferric Sulfate Pickle:

Sulfuric acid	13 oz./gal.
Ferrisul*	13 "

Temperature: Low copper alloys—120-140°F.
High copper alloys—140-175°F.

After pickling and prior to bright dipping, preliminary smoothing of the surface is often employed in order to increase the life of the bright dip and to obtain a more uniform finish. The solution is called a "Scaling" or "Fire-Off" dip and is composed of the following:

4. Scaling Dip:

Sulfuric acid	2 gal.
Nitric acid	1 "
Water	5 "

Agitate the work. If action is too rapid increase the sulfuric acid content.

BRIGHT DIPPING

Bright dips for copper and its alloys consist of nitric and sulfuric acids in widely varying proportions together with a small quantity of water and hydrochloric acid. A typical formula will consist of:

1.

Sulfuric acid	2 gal.
Nitric acid	1 "
Water	1 qt.
Hydrochloric acid	½ fl. oz.

Room temperature.

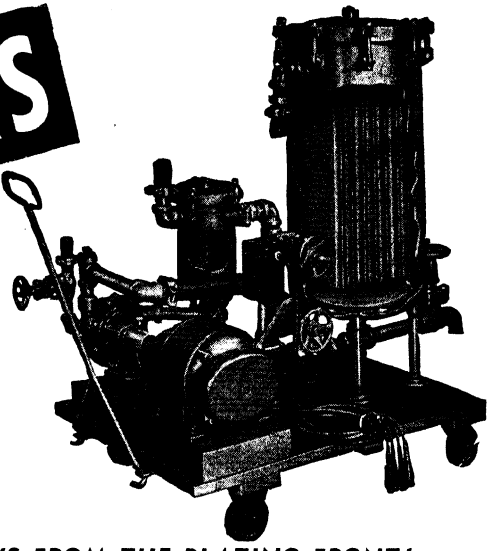
The hydrochloric acid is added to increase the lustre but an excess will tend to cause spotty surfaces. Wood soot and activated carbon are often added,

FILTERS

for

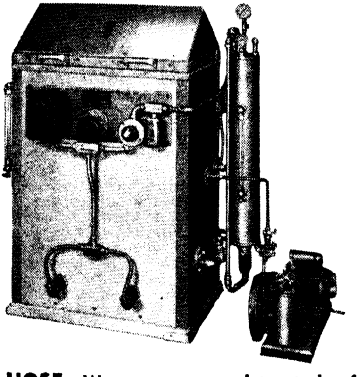
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especially when excessive amounts of hydrochloric acid are present, and tends to improve the operation of the dip. Sodium chloride may be used in place of hydrochloric acid in the formula.

If the work is coated with scale, treatment in a pickling solution or "scaling" dip is advisable before bright dipping. After bright dipping and rinsing thoroughly in cold, running water, articles may be dipped in a solution of 4 oz./gal. sodium cyanide to remove stains and, after an additional cold rinse, in a hot water rinse containing from ⅛ oz./gal. of soap, if the articles are to be lacquered, to 1 oz./gal. if no further treatment is required.

Tanks should be water jacketed to keep the temperature of the bright dip from rising and may be made of stoneware, 18-8 stainless steel and metal coated with suitable organic materials. Dipping baskets may be of stoneware, aluminum, 18-8 stainless steel or Nichrome, preferably the latter two, since aluminum is attacked by alkaline cleaners and stoneware is rather sensitive to the thermal shock involved in rinsing after cleaning.

Where a semi-bright finish is satisfactory the following dichromate dip will be found to be less expensive and more convenient to handle than the nitric-sulfuric bright dip, since no cooling is required and there are no objectionable nitrous fumes. Lead or lead lined equipment is used.

2. Sulfuric acid 12 oz./gal.
 Sodium dichromate 4 "
 Room or elevated temperature.

MATTE DIPPING

A solution which produces a light matte finish on copper and its alloy consists of:

1. Sulfuric acid 1 gal.
 Nitric acid 1 "
 Zinc oxide or sulfate 2 lbs.

This solution is operated hot and must be kept free of water. The finish produced in this dip is coarsened by adding nitric acid and made finer by adding sulfuric acid.

A very dull finish, such as is used for Army enlisted men's buttons, is produced by etching at room temperature in:

2. Hydrochloric acid 1 gal.
 40° Ferric chloride sol'n 1 "

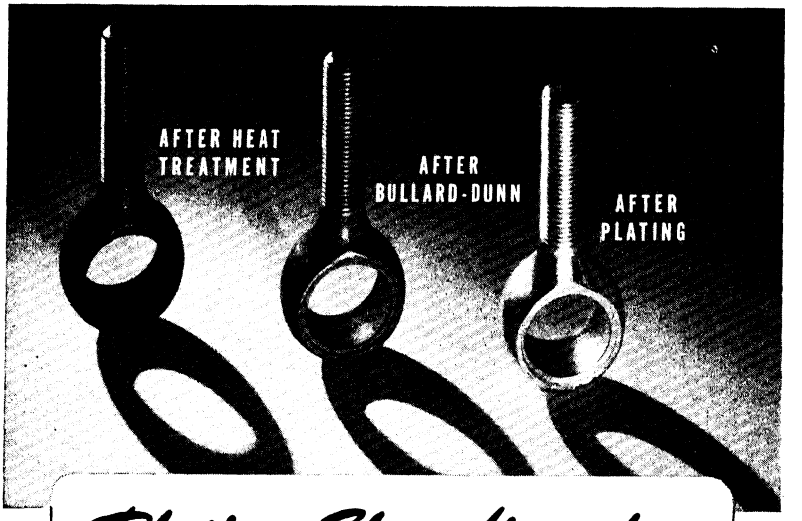
When etched to the required degree, the articles are rinsed in cold water and given a short immersion in a dichromate bright dip, such as given above, in order to remove smut.

IRON AND STEEL

Removal of scale and rust from iron and steel is accomplished ordinarily by the use of sulfuric or hydrochloric acid. At room temperature the action of hydrochloric acid is more rapid than sulfuric acid and is therefore, preferable for light oxide films, such as are found on polished steel. Sulfuric acid has the advantage of lower cost, however, and can be heated in a lead lined tank with lead steam coils, lead pipe immersion gas heaters or lead sheathed electric units, whereas

ELECTROLYTIC DESCALING

Electrolytic descaling is faster and gives greater assurance of thorough scale removal. The Bullard-Dunn Process is the only electrolytic method, however, that does not attack the work.



Plating Shops find that THE BULLARD-DUNN PROCESS is Tops for them... because

1. *It removes all kinds of oxide from all kinds of shapes and sizes of parts.*
2. *It is fast — no hand wiping necessary.*
3. *It does not attack the work.*
4. *It produces the cleanest surface possible for plating.*
5. *It is economical.*
6. *It is simple to operate.*

BULLARD-DUNN PROCESS DIVISION

OF THE BULLARD COMPANY

BRIDGEPORT 2, CONNECTICUT

hydrochloric acid requires a carbon or tantalum coil or sheathing and tanks with rubber, brick or suitable organic linings which are more expensive.

Pickling inhibitors are in universal use with these acids, to minimize attack on the metal during removal of the scale or rust. They conserve acid and reduce pitting of the metal to a minimum. Other addition agents are available for obtaining uniform pickling action by lowering the surface tension. Details regarding suppliers of such addition agents may be obtained from *Metal Finishing.*

In addition to hydrochloric and sulfuric acids, various other materials are used for certain requirements and some typical formulas are given below.

CAST IRON

1. Sulfuric acid 1 pt.
 Hydrofluoric acid 1 "
 Water 1 gal.
 Temperature: Room or elevated.

2. Sulfuric acid 12 oz.
 Nitric acid 5 "
 Zinc 1 "
 Water 1 gal.

IRON AND STEEL

1. Hydrochloric acid 1 gal.
 Water 1 "
 Room temperature.

2. Sulfuric acid ½ pt.
 Water 1 gal.
 Temperature: Room to 175°F.

3. Sulfuric acid 3 oz./gal.
 Potassium nitrate 3 "
 Temperature: 160°F.

4. Sulfuric acid 6 oz./gal.
 Ferrisul* 5 "
 U. S. Pat. 2,295,204.

5. Citric acid 10 oz./gal.
 Ammonia is added until the solution is alkaline to litmus. Used at boiling point to remove rust without any measurable attack on the metal.

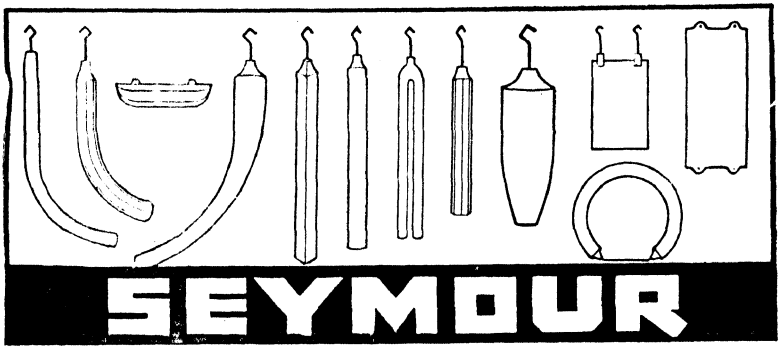
6. Phosphoric acid 10-15%
 Water 90-85%
 Temperature: 175-185°F.

7. Sulfuric acid 8 fl. oz./gal.
 Hydrochloric acid 10 " "
 Room temperature.

MAGNESIUM AND ITS ALLOYS

1. Chromic acid 24 oz./gal.
 Temperature: 190-212°F.

The following dips are rapid but remove sufficient metal to change the dimen-



NICKEL ANODES

99%+ Rolled Depolarized—A very fine, homogeneous, even-grained, virtually pure nickel anode. Corrodes in hot or cold solutions with minimum metallic loss. Forms practically no sludge. Especially suitable for baths having a pH above 4.5 electrometric.

99%+ Cast Oxide Depolarized — Almost entirely pure nickel. Suitable for most cold nickel baths, but especially those having a high pH. Corrodes evenly under most ordinary shop conditions.

99%+ Rolled Carbon—A rolled anode of high efficiency for low pH hot Watts bath not above 4.5 electrometric. Corrodes smoothly with a minimum of loose nickel.

99%+ Cast Carbon—A pure nickel anode with carbon content adjusted for smooth, uniform corrosion without special controls. Available, however, with carbon content to meet any specifications. Adaptable for general shop work using hot or cold baths.

"Seycast"—An improved type of 99%+ cast anode of very high efficiency, designed primarily for Bright Nickel baths, but works perfectly in any hot Watts bath having a pH of 4.5 electrometric or lower. Corrodes evenly with practically no loose nickel formation, due to long, interlocking grains radiating from a common center. Especially suitable for high current densities.

Bright Nickel—A hot organic type process free from promoter metal, stable and easily controlled. Produces brilliant deposits from a standard hot Watts bath without the usual coloring or buffing. Excellent throwing power. Wide operating conditions. Free from wetting out agents.

OTHER ANODES

Seymour Anodes in all shapes to meet rigid specifications are also available in copper, brass, bronze and zinc. The copper used is electrolytic. The zinc is always the best grade obtainable.

THE SEYMOUR MANUFACTURING CO.

60 FRANKLIN STREET **SEYMOUR, CONN.**

sions so that they should be used only *before* machining.

2. Nitric acid 10 fl. oz./gal.
 Sulfuric acid 3 " "
 Room temperature.

3. Chromic acid 16 oz./gal.
 Nitric acid 1 pt./gal.
 Room temperature.

NICKEL AND ITS ALLOYS

INCONEL

1. Water 2 gal.
 Nitric acid 1 "
 Hydrofluoric acid 1 pt.
 Temperature: 150-165°F.

2. Sulfuric acid 13 oz./gal.
 Rochelle salt 13 "
 Temperature: 160-180°F.

MONEL AND NICKEL

1. Water 2 gal.
 Hydrochloric acid 1 "
 Cupric chloride ½ lb.
 Temperature: 180°F.

The cupric chloride accelerates scale removal but may be omitted. After pickling in the above mentioned solution, rinse in hot water and immerse in the nitric-sulfuric acid or dichromate dip used for copper alloys.

For light scale, such as is found on work annealed in a reducing atmosphere, or for small, hot-worked parts the following may be used:

2. Sulfuric acid 1 pt./gal.
 Sodium nitrate ¾ lb./gal.
 Sodium chloride ¾ "
 Temperature: 180-190°F.

3. Sulfuric acid 13 oz./gal.
 Ferrisul* 24 "
 Temperature: 180°F.

4. Hydrochloric acid 13 oz./gal.
 Ferrisul* 13 "
 Temperature: 160-180°F.

NICKEL SILVER

1. Water 5 gal.
 Sulfuric acid 1½ lb.
 Sodium dichromate ½ "
 Temperature: Room or elevated.

2. Sulfuric acid 13 oz./gal.
 Ferrisul* 13 "
 Temperature: 140°F.



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SILVER

1. Nitric acid 2 gal.
 Water 1 "
 Temperature: Room or elevated.

2. Sulfuric acid 1 gal.
 Nitric acid 1 pt.
 Temperature: Room or elevated.
 Work should be dry.

3. Water 1 pt.
 Sodium cyanide 2 oz.
 100 vol. hydrogen peroxide ½-1 oz.
 Room temperature.
 This should be placed in a sink since it may boil over when work is immersed. It must be made up fresh.

4. Anhydrous alum 7 oz./gal.
 Sodium cyanide 4 "
 Soda ash 1½ "
 Temperature: 180-210°F.

STAINLESS STEEL

Since forging and annealing scales which form on stainless steel are very resistant to acids, it is often advisable to pickle in two operations, the first for loosening the scale and the second for removal or for brightening the surface. Where only light scales are present, the scale loosening treatment may be omitted. Suitable inhibitors should be added to minimize pitting of the metal.

SCALE LOOSENING

1. Sulfuric acid 12 oz./gal.
 Temperature: 180°F.

2. Sulfuric acid 1 gal.
 Hydrochloric acid 1 "
 Water 8 "
 Temperature: 130-140°F.

SCALE REMOVAL

1. Nitric acid 1 gal.
 Hydrofluoric acid 1½ "
 Water 2½ "
 Temperature: 125-150°F.

2. Sulfuric acid ½ pt./gal.
 Hydrofluoric acid ½ "
 Chromic acid 8 oz./gal.
 Temperature: Room or elevated.
 U. S. Pat. 2,172,041.

3. Nitric acid 1 gal.
 Hydrofluoric acid 1 qt.
 Hydrochloric acid 1 pt.
 Water 9 gal.
 Temperature: 130-140°F.

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4. Sulfuric acid 7-10 lbs.
 Nitric acid 12-15 "
 Hydrofluoric acid 1½- 2 "
 Water To make 100 lbs.
 U. S. Pat. 2,337,062.

5. Ferrisul* 8 oz./gal.
 Hydrofluoric acid 1½-2 "
 Temperature: 140-180°F.

6. Hydrochloric acid 5 gal.
 Nitric acid 1 "
 Water 14 "
 Temperature: 125-160°F.

PASSIVATION

1. Nitric acid 1 gal.
 Water 4 "
 Temperature: 120°F.
 Time: 20 minutes.

2. Nitric acid 1 gal.
 Water 1 "
 Temperature: Room.
 Time: 1-2 hours.

Passivation or immunization, as it is sometimes called, should be employed after any pickling operation on stainless steel. Effectiveness of this treatment may be tested by application of copper sulfate solution, slightly acidified. No copper will be deposited on passivated surfaces.

ZINC AND ITS ALLOYS

Bright finishes may be produced on zinc and zinc base alloys by immersion in chromic acid solutions containing chlorides and sulfates. Examples are as follows:

1. Chromic acid 30-40 oz./gal.
 Sodium sulfate 2- 4 "
 Room temperature.

Articles are immersed in the above mentioned solution for 5 to 30 seconds and rinsed in cold water. If a yellow film remains on the surface, it may be removed by immersion in a solution of about 1 oz./gal. of sulfuric acid.

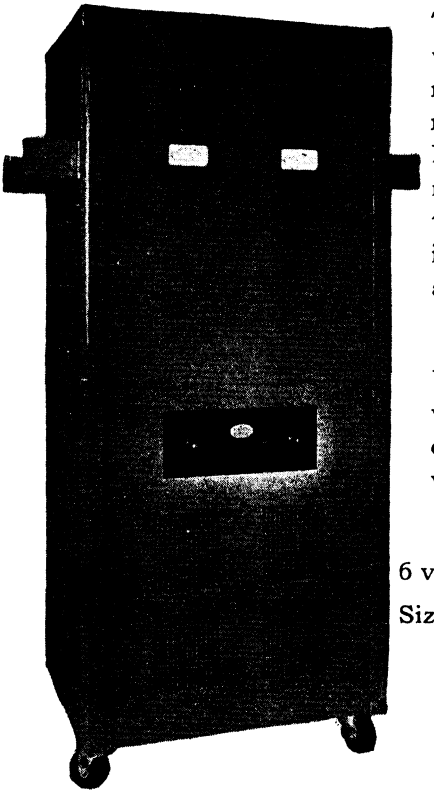
2. Chromic acid 25-60 lbs.
 Hydrochloric acid 10 "
 Water To make 100 lbs.
 Room temperature.

The above dip should contain at least 15% chromic acid and not more than 15% hydrochloric acid. Articles are immersed for 1 minute and rinsed. The surface has a dirty brass color which is removed by immersion for 1 minute at room temperature in a 10-70% chromic acid solution.

U. S. Pat. 2,125,458.

*Ferrisul is the trade name for commercial anhydrous ferric sulfate, produced and sold by the Merrimac Division of Monsanto Chemical Company, Everett Station, Boston, Massachusetts. Uses have been patented. The product contains a minimum of 90% ferric sulfate.

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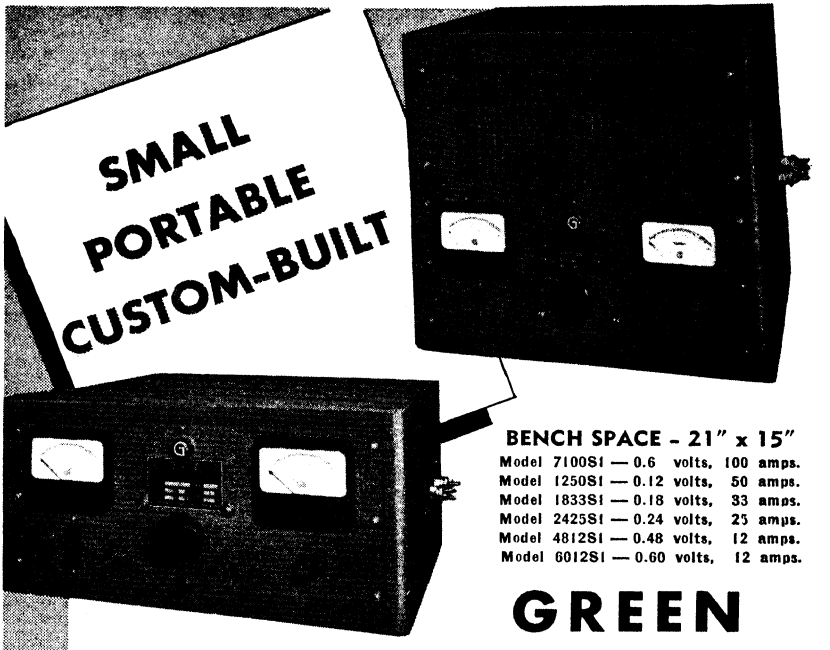
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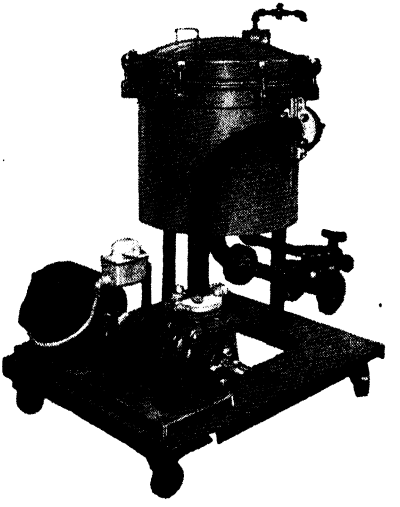
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Write for Bulletin 299



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SPARKLER

ENGINEERED FILTRATION

PLATING SOLUTIONS

ARSENIC PLATING

By G. B. Hogaboom, Jr. and Nathaniel Hall

White arsenic, As_2O_3	16 oz.	Temp.	Room temp.
Caustic soda	16 "	Current density	3-20 amp./ft.^{2}
Sodium cyanide	½ "	Voltage	2¼-4 volts
Water to make	1 gal.	Anodes	Steel

The arsenic content of this solution is about 12 oz./gal. and the free caustic soda, determined by the Sulfo-Orange method is about 4.5-5.0 oz./gal.

Addition of ½-1 oz./gal. of copper cyanide, dissolved in a minimum amount of sodium cyanide, will result in a darker color. The deposit in this case will be smutty and easily relieved, making it suitable for antique finishes.

A steel tank may be used as the container and may be made the anode.

BRASS AND BRONZE PLATING

By G. B. Hogaboom, Jr. and Nathaniel Hall

BRASS

Copper cyanide	4 oz.	Metallic copper	2.8 oz.
Zinc cyanide	1¼ "	Metallic zinc	0.7 "
Sodium cyanide	7½ "	Free cyanide	2 oz./gal.
Sodium carbonate	4 "	Temp.	75-100°F.
Water	1 gal.	Current density	3-5 amp./ft.^{2}
		Voltage	2-3 volts
		Anodes: brass; 80% copper, 20% zinc	

In making up a new solution it is often advisable to use about 6.5-7 oz./gal. of sodium cyanide instead of 7.5 oz. in order to get increased cathode efficiency.

One pint of ammonia added to each 100 gallons will aid in producing a good color when starting a new solution. It is possible to operate a brass solution at room temperature, but a more uniform and clearer color will be obtained if the temperature is maintained at some point near 100°F. It is absolutely essential, if a uniform color is desired in the operation of a brass solution, to hold all conditions within reasonable limits from day to day. The deposition of zinc is favored if the free cyanide increases, or the current density increases, or if the temperature decreases when the same current conditions exist.

Arsenic is used as a brightener. Dissolve 2 lbs. of caustic soda in ½ gal. of water and then add 1 lb. of white arsenic. Use 2-4 fluid oz. of this solution to each 100 gallons of plating solution. An excess must be avoided as it will cause the anodes to turn black and the brass deposit will have a very pale color.

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BRONZE

Copper cyanide	4.0 oz./gal.
Zinc cyanide	0.3 "
Sodium cyanide	5.0 "
Rochelle salts	2.0 "
Metallic copper	2.1 "
Metallic zinc	0.17 "
Free cyanide	0.5 "
Temperature	75°-100° F.
Current density	2-4 amp./ft.^{2}
Voltage	2-3 volts

Anodes, brass; 92% copper, 8% zinc.

The bronze solution is similar to a brass in that it deposits an alloy of copper and zinc. In a brass solution, a ratio of 80% copper to 20% zinc is desired. In bronze deposition, the proper ratio is 92% copper, 8% zinc.

Rochelle salts are added to improve anode corrosion. Arsenic or ammonia should not be used. The production of a uniform bronze color requires even more careful control of conditions than is the case with a brass solution. Operating at room temperature will produce a light bronze, while a higher temperature will produce a red bronze.

COPPER-CADMIUM BRONZE

A bronze solution in which cadmium is substituted for the zinc may be used for a bronze deposit which is more uniform in color and is easier to control since the anodes do not polarize so readily. The solution contains:

Copper cyanide	3 oz./gal.
Cadmium oxide	1/8-1/4 "
Sodium cyanide	4.5 "
Soda ash	2 "
Metallic copper	2.1 "
Metallic cadmium	0.11-0.22 "
Free cyanide	1.0 "
Temperature	Room temp.
Voltage	2-3 volts
Anodes	Copper

The cadmium which is plated out is replaced by small additions of a concentrated solution of cadmium oxide in sodium cyanide, when the deposit becomes copper colored or reddish. An excess of cadmium causes the deposit to become white. The excess is removed by plating out on dummies or by additions of sodium sulfide.

CADMIUM PLATING

By R. O. Hull

R. O. Hull & Co., Cleveland, Ohio

Cadmium plating is conducted almost exclusively through the medium of cyanide plating baths containing organic and frequently inorganic addition agents, necessary for production of semi-bright to bright deposits. Although cadmium cyanide plating baths are simple to operate and maintain for production purposes in which the current and thickness of deposit are not important, proper operation for production of good quality deposits of pleasing appearance and adequate thickness necessitates close control of solution composition and operating conditions similarly to almost every other plating bath. For this reason, even though a solution may appear to be operating correctly, it cannot be too strongly recommended to analyze and adjust the solution at regular intervals for continuous, good performance. The obvious result of inadequate solution control may be the production of burned or dull deposits, or improper coverage of work, while the less obvious but perhaps more important result may be diminution of cathode efficiency and hence production of inadequate thickness of deposit for good rust protection.

SOLUTION COMPOSITION

Typical solutions may be prepared using the following formulas for both still tank and barrel plating:

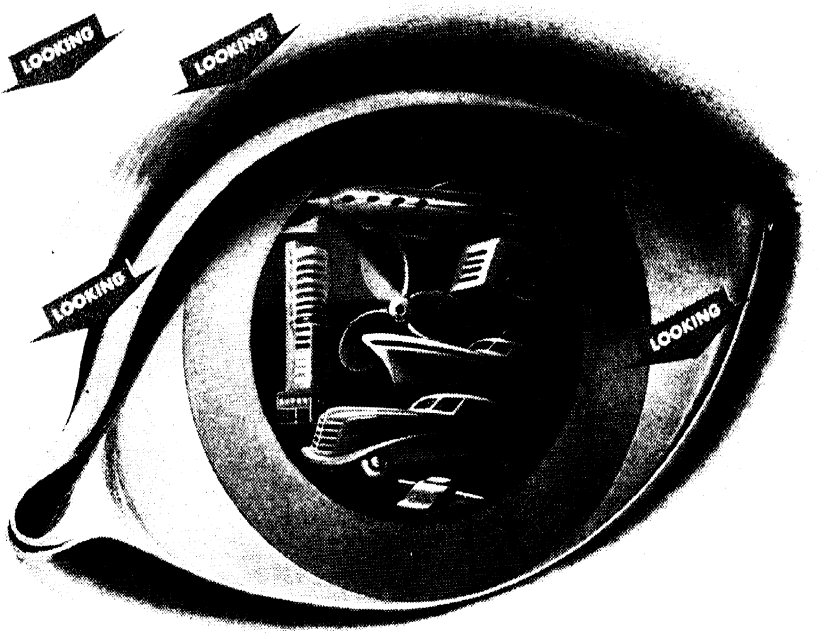
Cadmium oxide	3.5 oz./gal.
Sodium cyanide	14.5 "
Brightener	as required

OPERATING CONDITIONS

Temperature	70-95°F.
Current density	15-45 amp./ft.^{2} in still tanks
	5-25 amp./ft.^{2} in barrels
Voltage	1 to 4 volts in still tanks
	7 to 14 volts in barrels
Anodes	Cadmium bars or balls

MAINTENANCE AND CONTROL

1. Although quite wide ranges of composition may be used, it is necessary to maintain the *ratio* of total sodium cyanide to cadmium at about 3.8 to 4.2 to maintain high cathode efficiency of solution and to minimize the rate of sodium carbonate formation. Lack of attention to proper addition of sodium cyanide results in low anode efficiency and depletion of the cadmium content of solution which simultaneously results in rapid accumulation of sodium carbonate due to cyanide decomposition. Improper attention to solution composition may have no immediate effect but always results in a solution that eventually becomes inoperable. The most important addition is therefore sodium cyanide, which should be made at intervals of at least twice a week and preferably once each day. It is possible at the present time to use proprietary mixtures of sodium cyanide and brightening agents compounded in such proportions that addition of sodium cyanide as needed in the average solution also adds the proper proportion of the brightening agents. This is of practical importance since sodium cyanide is deter-



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But plating's future is secure. Solutions to *today's* plating problems exist because platers planned them . . . yesterday.

Though plated products change, this necessity for planning ahead is ever with us. That's why "looking ahead with platers" *has been* a major activity with H-VW-M since plating's infancy, why it will continue to be. That's why all branches of the plating industry have come to regard H-VW-M not merely as a manufacturer or supplier of equipment, but as a *co-worker*, willing and able.

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SINCE 1820

mined by chemical analysis, whereas organic addition agents cannot readily be determined.

2. The second item of importance is maintenance of adequate anode area in solution, and it is highly important that the amount of cadmium anodes in the solution be maintained to at least two pounds per gallon at all times as determined by daily inspection. If cadmium anodes are maintained properly, there should be little occasion for addition of cadmium oxide or cadmium hydrate unless dragout losses are unusually high. Analysis for cadmium metal should be made at least once a week and the solution adjusted to the proper composition, if necessary by addition of cadmium oxide or hydrate.

3. A small amount of sodium carbonate in cyanide cadmium is beneficial to solution operation, and the sodium carbonate should be maintained between the limits of 2 to 6 ounces per gallon. Sodium carbonate accumulates slowly in a properly maintained cadmium solution, but should be checked about every three months by analysis, and reduced to 2 ounces per gallon. Excessive sodium carbonate results in a low bright current density range and a high dragout rate. If the sodium carbonate is present in excess of 8 ounces per gallon, it can be reduced to this concentration by "freezing out" or cooling to a temperature of about 25°F. Additional removal of carbonate is best accomplished by means of a special gypsum manufactured for this purpose.[2]

4. Caustic soda in cadmium plating solutions should be present for production of good deposits and for proper conductivity of solution. It should be remembered that addition of cadmium oxide or hydrate is equivalent to caustic soda addition, each ounce per gallon of cadmium oxide or hydrate being equivalent to 0.6 oz./gal. of caustic soda. The pH as checked by pH papers or by the high pH glass electrode should be approximately 13, corresponding to a free caustic soda content of 1.5 to 3.2 ounces per gallon. If caustic soda is found by analysis to be less than 1.5 ounces per gallon, appropriate addition should be made to raise it to 2.5 ounces per gallon by cadmium oxide or hydrate or caustic soda.

5. Although it is possible to produce consistently bright deposits from modern cadmium plating solutions containing the proper addition agents, the uniformity of coating and tarnish resistance can be improved by means of a bright dip following plating. The bright dips in general use are of three types, i.e., nitric acid[3], peroxide[4], or chromic acid[5].

EQUIPMENT

For general purposes, either rubber-lined or steel tanks may be used for cyanide cadmium, although the former offers advantages in elimination of stray current losses. Cylinders for barrel cadmium plating may be either of hard rubber or of Bakelite. Regardless of material of construction, it is necessary to keep cadmium plating solutions within reasonable limits of temperature, if consistently bright deposits are to be expected and for this purpose, a cooling coil is necessary. For barrel plating, the cylinder should never be rinsed, but the work from the barrel should be dropped into a suitable basket or tray. Rinsing of the barrel results in exceedingly high dragout losses and is not only expensive through solution replacement but makes maintenance of solution difficult.

ADDITION AGENTS

For production of bright deposits, inorganic addition agents may be used in conjunction with organic addition agents [see 1]. Organic addition agents for cadmium plating may be of many types, most of which are the subjects of patents.

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East: Safety and Maintenance Co., Inc., 601 W. 26th St., New York 1, N. Y.

Since the most effective addition agents are used in proprietary processes, detailed recommendations cannot be given.

RATE OF DEPOSITION

Cadmium can be deposited at fairly high current densities and efficiencies and since it has a high atomic weight with a density about that of nickel, deposits are produced rapidly. For still plating, it is safe to assume a deposition rate of 0.0003" in 10 minutes at 20 amp./ft.^{2} but in barrel plating, 30 minutes should be allowed for the same thickness with an average voltage of 11-13 across barrel and anodes. Other thicknesses, plating times, and current densities can be calculated from these conditions, although this thickness is the minimum for a good corrosion resistant deposit. (These rates are given as typical and they may vary with different types of solution and plating conditions. When plating to specifications, always check the thickness of plate obtained and do not rely merely on time-current density factors.—Ed.)

1. Organic and frequently inorganic addition agents are necessary for bright deposits.
2. U. S. Pat. No. 2,164,924.
3. U. S. Pat. No. 1,816,837.
4. U. S. Pat. Nos. 2,154,468 ; 2,154,469 ; 2,154,455 ; 2,154,451.
5. U. S. Pat. Nos. 2,021,592 ; 2,070,871 ; 2,186,579.

TREATMENT OF METALS FOR HARD CHROMIUM PLATING

By Arthur Logozzo

Nutmeg Chrome Plating Co., Hartford, Conn.

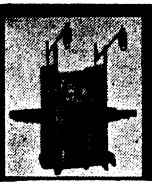
Inasmuch as most parts to be hard chromium plated are either just machined or ground, we are not confronted with a pickling problem but one of surface preparation. This is all-important because of the direct relationship between the basis metal surface and the nature of the chromium deposit.

The basis metals can be classified into four groups: the carbon steels, nickel-chromium alloys, cast irons and carbides. None of these groups of metals are particularly difficult to plate, regardless of the thickness requirements, if some simple rules, as laid out in the following tables are followed, plus the use of common sense.

It will be noted in the case of cast irons that two methods are listed—this in the event the first one does not produce the desired results. No trouble will be encountered, however, unless the material is particularly poor, in which case the second method is generally fool-proof.

CLEANING AND ETCHING PROCEDURES

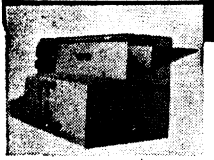
Type of Steel	Alkaline Treatment	Etching Medium	Thickness of Deposit	Etching Time
Low Carbon	Anodic	Anodic	0.0002"	½- 1 minute
	16 oz./gal.	Chromic acid	0.001"	2- 4 minutes
	6 volts	28 oz./gal.	0.005-0.010"	5-10 minutes
High Carbon	Anodic	Anodic	0.0002"	15-30 seconds
	16 oz./gal.	Chromic acid	0.001"	1.5- 3 minutes
	6 volts	28 oz./gal.	0.005-0.010"	3- 5 minutes



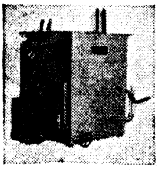
OPTIMUS Jet Type Parts Washing and Rinsing Machine.

A Complete Line

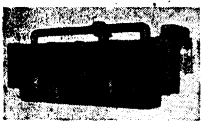
to meet your metal washing, rinsing and drying needs, *before* and *after* plating



OPTIMUS Screw-Drum Type Machine for washing, rinsing or drying.



OPTIMUS Spray Type Parts Washing and Rinsing Machine for handling Parts on racks.



OPTIMUS Continuous Type Washing Machine for handling parts on racks.



OPTIMUS Continuous Type Cold and Hot Air Dryer for handling parts on racks.

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The result is a limited number of models of proven design that cover a wide number of conditions. A glance at this table shows small number of basic type of units (7) and the large complete range of conditions (12) met:

BEFORE PLATING	In Baskets		Single Baskets Baskets on Conveyor	Model No. A-1 Model No. B-1
	Rotary Drum Washing of small parts			Model No. E-1
	After Racking		Single Racks Racks in Bulk	Model No. C-1 Model No. D-1
AFTER PLATING	After Barrel Plating		Rinsing Rinsing & Drying Drying	Model No. E-2 Model No. E-6 Model No. E-3
	After Plating On Racks	Single Rack	Rinsing Drying	Model No. C-2 Model No. C-3
		Continuous	Rinsing Drying	Model No. D-2 Model No. D-3

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Moly	Anodic 16 oz./gal. 6 volts	Anodic Chromic acid 28 oz./gal.	0.0002" 0.001" 0.005-0.010"	1 minute 5- 6 minutes 10-15 minutes
Nickel-Chromium Alloys	Immersion soak only	Anodic Chromic acid 28 oz./gal.	0.0002" 0.001" 0.005-0.010"	½- 1 minute 2- 3 minutes 5 minutes
Moly	Anodic 16 oz./gal. 6 volts	Sulfuric Acid	0.0002" 0.001" 0.005"	½ minute 1- 2 minutes 2- 3 minutes
High Speed	Immersion soak only	Anodic Chromic acid 28 oz./gal.	0.0002" 0.001" 0.005"	10-15 seconds 15-30 seconds 1- 2 minutes
Stainless	Cathodic clean	Chromic acid (Hydrochloric can also be used)	0.0002" 0.001" 0.005"	10-15 seconds 15-30 seconds 1- 2 minutes

CYCLE FOR SMALL TOOLS

(Drills, taps, reamers, etc.)

1. Heat treat at 600°-1200°F. (Consult metallurgist)
2. Anodic clean—12-16 oz./gal. at 6 volts.
3. Water rinse.
4. Immerse in 50% hydrochloric acid containing an addition agent.
5. Water rinse. This rinse must be thorough.
6. Plate—5 minutes average on new tools.
7. Bake—350°F. for 1-3 hours.

CYCLE FOR NON-FERROUS METALS

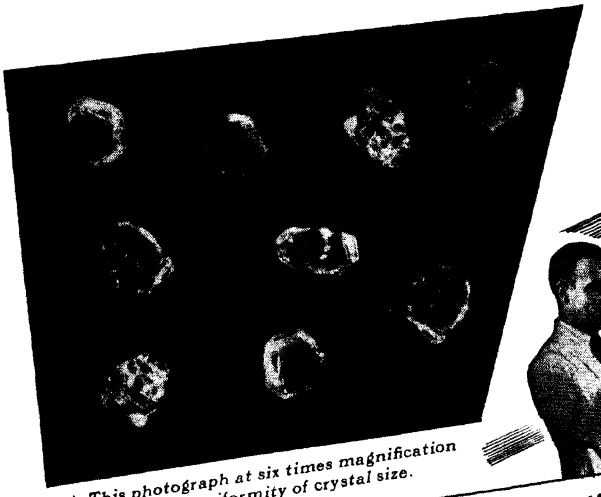
1. Cathodic or soak clean.
2. Water rinse.
3. Immerse in 50% hydrochloric acid or treat anodically in sulfuric acid.
4. Water rinse.
5. Plate.
6. Bake—200°-350°F. for 1-3 hours.

CYCLE FOR CAST IRON — METHOD I

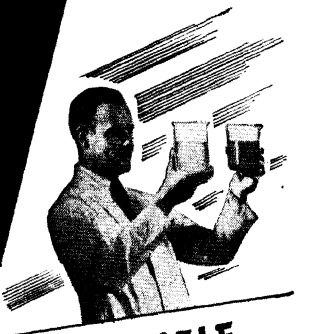
1. Anodic clean in alkaline cleaner.
2. Water rinse.
3. Etch anodically in chromic acid for 3-5 seconds.
4. Flash in plating tank at 10 amp./sq. in.
5. Drop current to normal 3-4 amp./sq. in. after approximately ½-1 minute.

CYCLE FOR CAST IRON — METHOD II

1. Buff well.
2. Wash in gasoline or organic solvent.
3. Dry very well.
4. Rub down entire surface using clean cloth and either slaked lime or aluminum oxide powder.
5. Flash in plating tank at 10 amp./sq. in.
6. Drop current to normal 3-4 amp./sq. in. after approximately ½-1 minute.



★ *This photograph at six times magnification shows uniformity of crystal size.*



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CYCLE FOR CARBIDES

1. Anodic clean in alkaline cleaner.
2. Water rinse.
3. Immerse in hydrochloric acid to activate surface.
4. Water rinse. This rinse must be thorough.
5. Place in plating bath and bring part to solution temperature.
6. Flash at 6 amp./sq. in. for 30 seconds.
7. Drop current to regular current density.

CHROMIUM PLATING

By Dr. George Dubpernell
United Chromium, Inc., Waterbury, Conn.

ORNAMENTAL CHROMIUM

When current is passed through a solution containing only pure chromic acid and water, no chromium plate results. To convert such a solution into a chromium plating bath requires the presence of acid radicals, such as sulphate, fluoride, etc., which are called "catalysts" because they permit the plating of chromium without themselves entering into the reaction.

The essential factor in bath composition in chromium plating is the ratio of chromic acid (CrO_3) to the total catalyst acid radicals expressed as sulphate (SO_4). For best results this ratio should be approximately $\frac{100}{1}$.

To supply sulphate to a chromium plating bath, either anhydrous sodium sulphate (Na_2SO_4) or sulphuric acid (H_2SO_4) can be used. Since sulphate is present in varying amounts even in the purest chromic acid, allowance must be made for it, as well as other acid radical catalysts, when adjusting the composition of the plating bath for maintenance in continuous operation. To supply fluoride which is used in some chromium plating baths, hydrosilicofluoric acid (H_2SiF_6) is often used.

A typical chromium plating bath composition is:

Chromic acid (CrO_3)	53 oz./gal.
Sulphate (SO_4)	0.53 "

It is important to ascertain the total catalytic effect of all of the various catalyst acid radicals present in an operating bath from whatever source, and to adjust the amount of these acid radicals so that it is equivalent in effect to 0.53 oz./gal. of sulphate in a bath containing 53 oz./gal. of chromic acid. When only sulphate is present in an operating bath, this is the only acid radical which need be taken into consideration. Frequently, however, other acid radicals, such as silicofluoride, are also present in chromium plating baths and when such is the case, their effect must also be taken into consideration when adjusting the ratio for optimum results.

To produce bright deposits from baths such as described above, the surface of the work to be plated should first be prepared so that it has the appearance desired for the finished surface and then plated, for example, at a temperature of 105°F. and a current density of 115 amperes per square foot. Coordination and



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- Preparing aluminum for plating.

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control of the important factors of (a) preparation of the base metal, (b) composition of the bath, (c) temperature, and (d) current density, are essential for successful results.

Solutions of lower concentrations of chromic acid can also be used, for example, 33 oz./gal. In such case, the total catalyst acid radical content of the bath must be adjusted so that its effect is equivalent to 0.33 oz./gal. of sulphate so as to maintain the ratio at its optimum of $\frac{100}{1}$. While baths containing 33 oz./gal. of chromic acid have a slightly higher current efficiency than those containing 53 oz./gal. of chromic acid, they have lower conductivity and, therefore, require higher voltage for a given current density. The more dilute baths are also more sensitive to the addition or subtraction of catalyst acid radicals from drag-in and drag-out and hence require more frequent and more careful adjustment for maintenance purposes. For instance, if a small amount of sulphate from a sulphuric acid dip is dragged into a chromium plating bath operated at 33 oz./gal. of chromic acid, it will cause more change in the ratio than in a bath operated at 53 oz./gal. of chromic acid.

7% tin-lead anodes are preferred for use in chromium plating baths. Antimony-lead anodes can also be used.

Trivalent chromium compounds and metallic impurities, such as iron, copper, and zinc, decrease the conductivity of the solution. When present in small amounts, their effect is inconsequential, but when these impurities are permitted to build up in large quantities they may be objectionable.

The processes described above for producing bright plate and the use of tin-lead anodes are covered by patents owned by United Chromium Incorporated, 51 East 42nd Street, New York City.

INDUSTRIAL OR HARD CHROMIUM

The industrial uses of chromium plate owe their existence to the unique combination of properties possessed by the deposited metal and not found in other methods of hardening. In addition to being hard, electrodeposited chromium has an especially low coefficient of friction, high seizure resistance, and high corrosion resistance when plated to sufficient thickness.

The best results are obtained in some cases with relatively thin chromium plate on a hardened steel foundation metal. Metal cutting tools with the edge properly prepared prior to chromium plating are examples of this. The thicknesses applied are generally about 0.00001" to 0.0005", and most commonly in the thinner part of this range. All of the chromium plate is frequently ground off one side of the cutting edge, leaving the deposit on the chip receiving side only, which gives considerably increased life to these tools in many types of service.

Heavier deposits of chromium are applied in other cases which may range upward in thickness to 0.010" and considerably more in special instances. Examples of this type of application are rolls and drums for various purposes, drawing dies and mandrels, plastic molds, gages, pump shafts and hydraulic rams. Generally 0.001" to 0.003" more chromium is deposited than desired, and the excess ground or polished off to give final dimensions and surface finish as desired.

The hardness of all bright chromium deposits is approximately the same (1000 to 1025 Brinell) and this type of plate is generally preferred for industrial chromium plating. A thin chromium deposit on a soft foundation metal such as copper or brass may not seemingly exhibit this hardness because it is easily gouged through and crushed due to lack of a proper backing. The same thin chromium

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deposit on a hardened steel foundation metal such as a cutting tool, may add appreciably to the hardness and durability of the tool. The following values for the Brinell hardness of different types of chromium plate are taken from an unpublished report in 1932 of T. H. Webersinn and J. M. Hosdowich of United Chromium, Inc., Research Laboratories:

Appearance or Type of Deposit	Brinell Hardness
Bright, as commonly used	1000 to 1025
"Burned" nodules, not usable	1250
"Milky" due to low current density	775
"Matte" or dull from cold solution	575

Some other physical properties of electrodeposited chromium taken from various sources and which may be of interest are:

Density (as deposited)	6.93
Density (after annealing)	7.10 to 7.15
Melting point	1920°C. (3,488°F.)
Electrical resistivity (annealed)	13.1 to 19.3 $\times 10^{-6}$ ohm/cm.^{3}
Reflecting power	60 to 66%
Coefficient of expansion (as deposited)	negative
Coefficient of expansion (annealed)	4.99 to 6.05 $\times 10^{-6}$
Coefficient of friction sliding on chromium	0.12
Coefficient of friction sliding on babbitt	0.13
Coefficient of friction sliding on steel	0.16

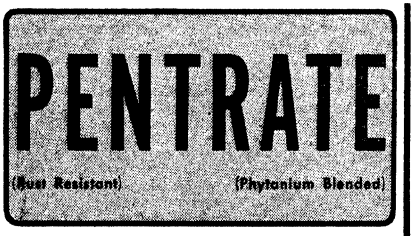
The solutions used for industrial chromium plating are the same as those for decorative plating and have been described above. The solution with about 33 oz./gal. (250 g./l.) CrO_3 and 0.3 oz./gal. (2.5 g./l.) SO_4 is very commonly used, and gives a plating speed of about 0.001″ thickness of chromium per hour at a temperature of 131°F. (55°C.) and a current density of about 2 amperes per square inch. The more concentrated solution containing about 53 oz./gal. (400 g./l.) CrO_3 and 0.5 oz./gal. (4 g./l.) SO_4 is also often used, and gives a plating speed of about 0.0005″ per hour at a temperature of 122°F. (50°C.) and a current density of about 1.5 amperes per square inch.

Cleaning before plating and the adhesion of the deposit are especially important for good results in industrial chromium plating. Aside from removing all grease, dirt, rust, and scale from the surface, good adhesion is usually assured by a reverse current etch in 50 to 55° Be sulfuric acid or in a chromic acid solution as the final step in the cleaning process. Generally the reverse current is applied for ½ to 1 minute with 6 volts, although shorter or longer times may be used in certain cases. Additional details on the preparation of surfaces for industrial chromium plating may be found in A.S.T.M. Specification B-177-43T, covering "Tentative Recommended Practice for Chromium Plating on Steel for Engineering Use."

High carbon steel, especially when hardened and of thin cross section, may be embrittled by chromium plating. This embrittlement can be largely removed by heating the part in an oven or oil bath at 300″F. to 500°F. for about an hour. Shorter or longer heating time may be used depending on the composition of the steel, the shape of the piece, and the use to which it is put. Such a heat treatment is desirable for most hardened steel tools.

In industrial chromium plating the use of insulation resistant to the action of the plating bath is especially important. Solid insulators are used for the con-

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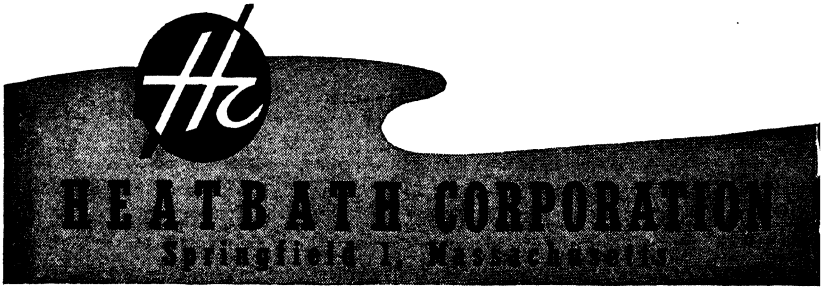
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struction of composite racks holding both the object to be plated and the anode in fixed relation to each other. Insulating lacquers are used on surfaces not to be plated, and insulated shields are used in a number of ways to control the flow of current and distribution and thickness of the deposit.

Some suggested hardness values for the steel foundation metal and thicknesses of chromium to be applied for various applications are given below from an unpublished report of M. Shacat of United Chromium, Incorporated:

Type of Article	Hardness of Steel (Rockwell C Scale)	Thickness of Chromium
Drills	C62 to C64	0.00005" to 0.0005"
Reamers	C62 to C64	0.0001" to 0.0005"
Burnishing bars	C60 to C62	0.0005" to 0.003"
Drawing plugs or mandrels	C60 to C62	0.0015" to 0.008"
Drawing dies	C62 inside C45 outside	0.0005" to 0.008"
Plastic molds	C55 to C60	0.0002" to 0.002"
Gages	C48 to C58	0.0001" to 0.0015"
Pump shafts	C55 to C62	0.0005" to 0.003"
Rolls and drums	----------	0.00025" to 0.012"
Hydraulic rams	----------	0.0005" to 0.004"
Printing plates (engraved steel)	----------	0.0002" to 0.0005"

POROUS CHROMIUM PLATING*

By Theodore G. Coyle

United Chromium, Inc., New York, N. Y.

Porous chromium plate is finding increasing use as a bearing surface, especially on the bores of cylinders for internal combustion engines, compressors, hydraulic equipment, piston rings, and other wearing parts where chromium plate is wanted for its superior wear resistance characteristics and its adaptability to salvage requirements. When the surface of the usual dense or hard chromium plate is broken up or "interrupted" to give a series or collection of indentations and prominences, it is called porous chromium plate.

METHODS

Porous chromium plate may be produced in a number of ways. It may be obtained by depositing a chromium plate over a cut, engraved, etched, grit-blasted or otherwise suitably indented or roughened surface; and then honing, grinding, lapping or polishing this chromium plate of finely irregular surface to give a plate made up of tiny peaks and valleys, or "plateaus" and "channels" with the peaks and plateaus flattened and smoothened by the finishing operation. The tiny plateaus, in aggregate, constitute the actual bearing surface; and the pits or valleys serve as wells or reservoirs for the lubricant. This method may be described as a "mechanical" method of producing porosity.

Porous chromium is also commonly obtained by the "chemical" method; i.e., etching the chromium plate itself, after deposition on a smooth basis surface.

*No reference is made in this paper to existing patents relating to porous chromium plated articles, their use, or methods of producing them.

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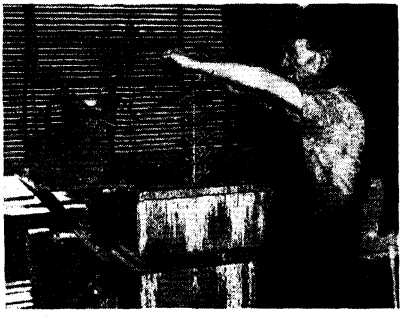


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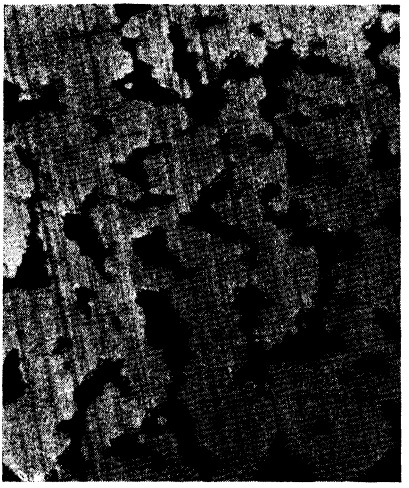


Fig. 1 x 30



Fig. 2 x 60

Here the plate, indented with pits or with a network of fissures or channels from the etching, is honed or otherwise finished smooth, as in the case of the plate deposited on the cut, etched, or grit-blasted basis metal, to give a chromium deposit having throughout its surface tiny plateaus and pits or crevices.

Porous chromium can also be obtained by a combination of the mechanical and chemical methods.



Fig. 3 x 30

Different types of porous chromium plate are shown in the accompanying photomicrographs. Figure 1 shows a "grit-blast" type porosity of the mechanical method, obtained by having a regular hard chromium plate deposited over a gritblasted surface. Figure 2 shows porosity by the chemical method of the "pitted" or "pin-point" type, obtained by honing the chromium plate etched after deposition. Figure 3 shows porosity also obtained by the chemical method and illustrates the "channel" type, obtained by etching and honing after deposition of the plate, as in Figure 2, but with the chromium plate deposited under conditions which "predisposed" it to development of large plateaus upon subsequent etching and honing.

The preferred "percentage porosity," or ratio of voids to total surface, is about the same for all porous chromium plate produced by the various methods,

and depends on the particular application. A range of from about 10% to 60% covers most applications, with a porosity depth of about 0.001 to 0.003 or 0.004 in. See Figure 4 which represents, schematically, the mechanical type porosity; and compare it with Figure 5, which represents porosity by the chemical method.

PROCEDURES

Mechanical Method

The procedure for porous chromium by the mechanical method requires first a proper engraving, etching, or grit-blasting of the basis metal to give a surface which,

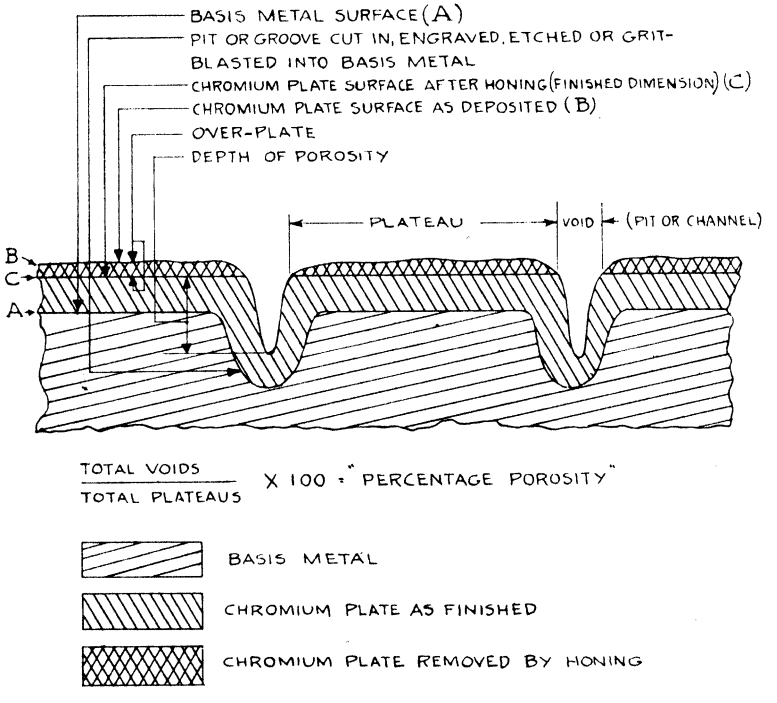


FIGURE 4. SCHEMATIC MAGNIFIED CROSS-SECTIONAL VIEW OF POROSITY BY MECHANICAL METHOD

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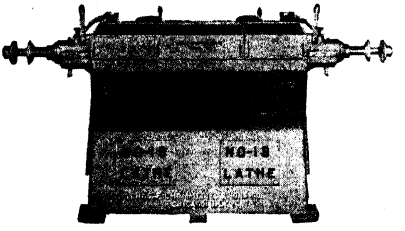
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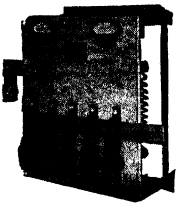
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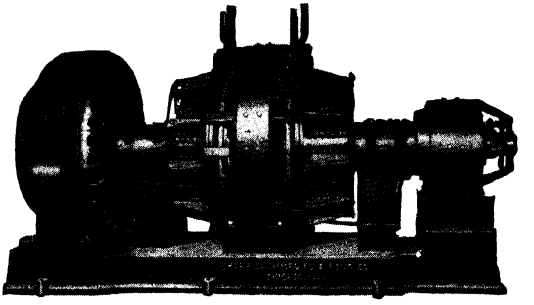
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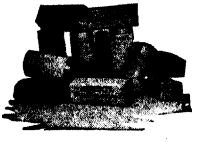
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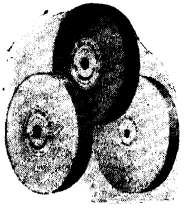
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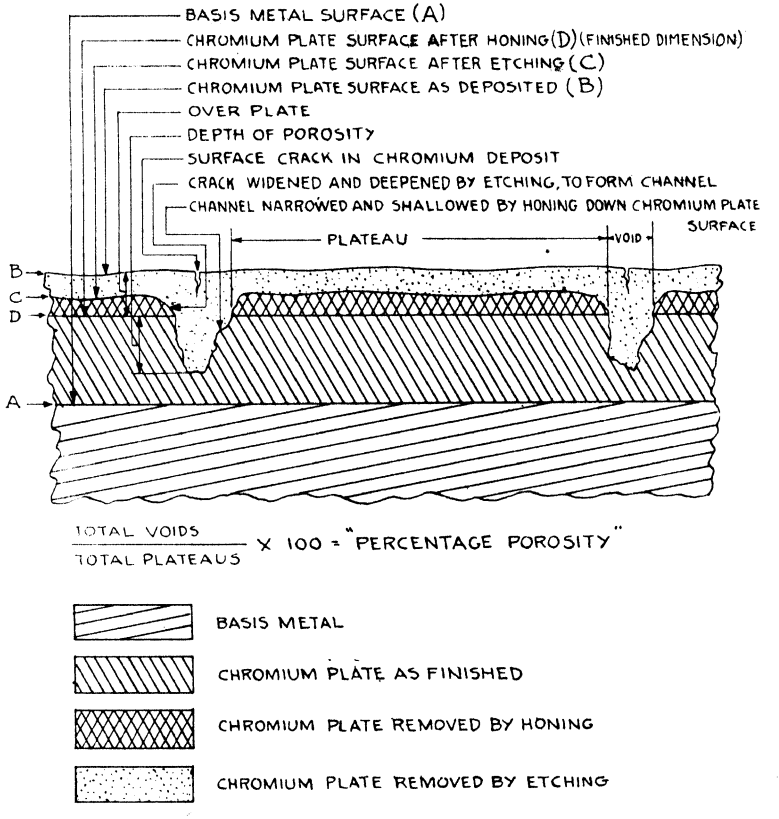


FIGURE 5. SCHEMATIC MAGNIFIED CROSS-SECTIONAL VIEW OF POROSITY BY CHEMICAL METHOD.

when chromium plated and honed, will result in a chromium surface of the desired percentage porosity, and one having plateaus of suitable size, uniformity, and distribution, and channels or pits of adequate depth. The chromium plating itself is done according to usual good practice for hard chromium plating, with cleaning methods suited to the basis metal being plated, and the racking especially adapted to requirements of uniform plate distribution.

With the mechanical method for porous chromium there is no critical minimum thickness of plate, other than the usual wear considerations; the thinner the plate, the more faithfully it reproduces the predetermined porosity pattern originally



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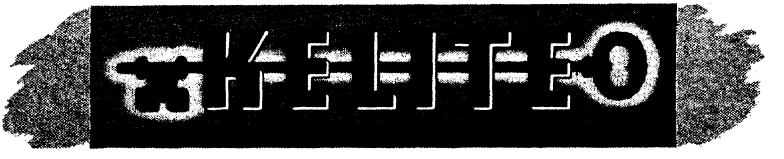
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put into the surface of the basis metal. As little as 0.001 in. of chromium or less would suffice in some instances although thicknesses of about 0.005 in. are more commonly used. The exact plate thickness selected would depend on the kind of pattern and degree of roughening of the basis metal as well as the application at hand. For all thicknesses, about 0.0005 to 0.001 in. "over-plate" is usually required to provide stock for honing back to desired porosity pattern at the plate thickness where correct finished dimensions are obtained.

In essence, the mechanical method consists of depositing a good, uniform hard chromium plate over a basis metal already etched or engraved to a predetermined roughness or porosity pattern, and honing or otherwise finishing this chromium plate to give smooth plateaus, while still retaining pits and indentations reproduced from the design in the basis metal.

Chemical Method

The procedure for producing porous chromium by the second and more commonly used method is referred to as the chemical method because it involves a chemical or electrochemical etching of the chromium plate itself, deposited generally on a smooth surface; i.e., it does not depend on any roughness or pattern in the basis metal. Here the pits, or a fissure network are etched into the chromium plate. The conditions under which the chromium plate is deposited determine the type and degree of porosity that can be developed on etching. The results on etching are controlled by the bath composition and operating conditions of the chromium deposition, and thus the plate can be deposited predisposed to a desired type of porosity development; for example, to pin-point or pitted porosity, or to channel or network porosity.

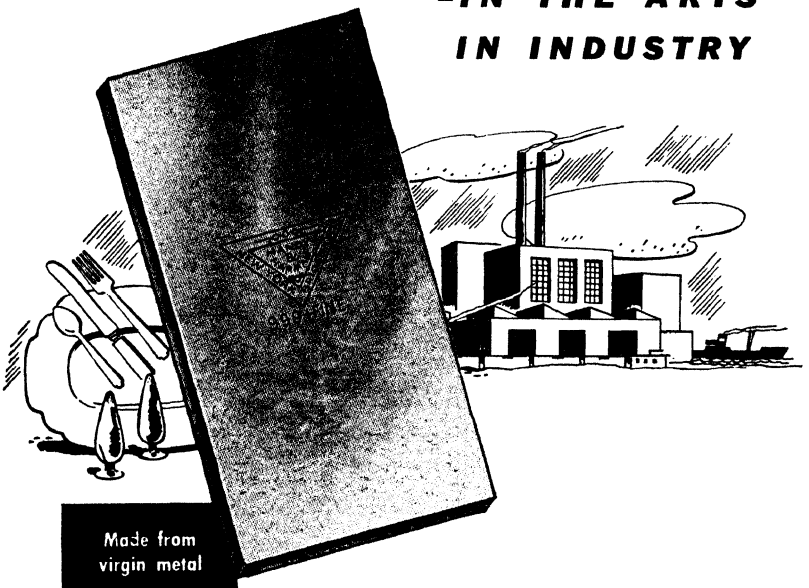
Except for the etching step and the need for special attention to plating solution conditions as they affect the deposited chromium itself, the technique in the chemical method is the same as that for the mechanical method; i.e., one uses the usual cleaning procedures according to the basis metal composition, good "hard chromium" plating technique and racking for uniform plate distribution, and honing, grinding, lapping, or polishing of the etched deposit for final development of the porosity to desired degree and dimension. The minimum plate thickness necessary is greater than with the mechanical porosity, since allowance must be made for some dissolving or stripping of the chromium on etching and also penetration of the pores into the plate short of reaching the basis metal; a minimum deposit thickness of about 0.004 in. is generally satisfactory. The maximum plate thickness is determined by the usual plating limitations but normal thicknesses run from 0.006 to 0.010 and 0.015 in. and higher. The requirements for uniformity of plate thickness apply here as in the case of the mechanical porosity and for the same reasons; i.e., limited depth of porosity and narrowing width of voids (reduction in percentage porosity) as plate is removed on honing.

It should be noted that with the chemical method, etching of the plate in some instances may be done after the plate has been honed or ground to size and shape, but the usual practice is to etch the plate just as deposited, for better porosity development. It is not uncommon, however, to re-etch a plate that has been honed after the initial etching, if insufficient porosity remains after the first honing operation. Such re-etching is generally followed by a second light honing operation to "clean up" the etched surface.

POROSITY CONTROL

The control of the porosity produced by the chemical method lies first of all in the plate itself; in addition, consideration should also be given to the etching and the final finishing after etching. The ultimate result desired, a good, well

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functioning porous chromium deposit, requires careful and proper regulation of these three factors: the plating, the etching, and the finishing; the first with respect to predisposition to a desired type of porosity, the second with respect to the amount of etching and the third with respect to the amount of plate removal in the finishing operation.

The Plating

The special attention to plating conditions required in the chemical method is due to the fact that chromium plate is predisposed to definite and different types of porosity development according to conditions of deposition. Deposited under one set of conditions, the plate when etched and honed will produce, for example, pitted or pin-point type porosity; under another set, channel or network type; under other conditions, channel type with different size of plateaus; and under still different conditions, other types intermediate to pitted and channel porosity. Since the type of porosity has been demonstrated to be of prime importance in the success of porous chromium plate for certain applications, it follows that the predisposition of the plate controlled by plating conditions is of fundamental importance.

The plating conditions which primarily control the predisposition of the deposit to type of porosity are two: the bath composition, with respect to chromic acid-catalyst acid radical ratio in particular; and the plating temperature. Current density, within limits, is of slight importance in predisposition of plate, although it is of course important with respect to controlling speed and plating accurately "to size" as is generally required in porous chromium plating. In the production of the channel type of porosity the control factors of ratio and plating temperature are closely correlated in controlling the size of the fissure network pattern obtained on etching. The plating conditions for the same porosity formation need not be identical, but these control factors must be correlated, one changed to compensate for a change in the other if predisposition to the same fissure network pattern is to be maintained. Within limits, an increase in ratio results in predisposition to a larger network pattern; and an increase in temperature also predisposes to an increase of the network pattern size.

While the same porosity result can be produced by various correlations of these control elements, actual practice has been largely standardized on two sets of conditions, one for pitted or pin-point type porosity, and the other for network or channel type porosity. In both cases, a 250 g./l. chromic acid bath is used, and a current density of 2½ to 3½ amp./sq. in. For the pitted type porosity a temperature of 122°F. and a ratio (CrO_3/SO_4) of 100/1 to 125/1 are employed. For the network or channel type porosity, a temperature of 140°F. and a ratio (CrO_3/SO_4) of 115/1 to 125/1 are used. Note that, while ranges are given here, each factor is narrowly held for any given set of operations. The temperature is maintained within plus or minus 1°F., preferably plus or minus ½°F.; and the ratio is kept as close as possible to the selected value by means of frequent bath adjustments.

The Etching

The etching may be simple chemical etching, using anything that will attack chromium plate; for example, hydrochloric acid. It may also be electrochemical, with cathodic treatment or anodic treatment; any one of these alone, or two or three in combination. The degree and control of etching is important however, and the electrochemical methods are well adapted to this requirement and widely used. In the electrochemical anodic etch, chromic acid is commonly used although other materials may be used; in short, anything that will dissolve the chromium. The extent of etching in the chromic acid anodic treatment, employing a chromic acid



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solution with or without sulphate, is usually measured in terms of ampere-minutes per sq. in. The temperature is not critical, and neither is the current density, which generally ranges from 2 to 4 amp./sq. in. The amount of etching may vary considerably depending on the result to be accomplished; that is, whether etching need be for porosity development alone or also for reducing plate thickness to come within dimension limitations after honing.

Cathodic electrochemical etching may also be used to develop porosity. The electrolytes used may be hydrochloric, sulphuric, phosphoric and oxalic acids, and acid and acid salt mixtures. Here, lower current densities are used, and excellent depth of porosity is obtained with minimum removal of chromium from plateau surfaces.

The straight chemical etching consists of simply letting a predisposed plate be attacked by the etching medium. This may be hydrochloric acid, or hydrochloric acid plus an accelerator, or hot dilute sulphuric acid.

The Finishing

In control of porosity, the final honing, grinding, or lapping is important from the standpoint of amount of plate removal; the more removed the smaller or lower the porosity for a given etching. With the channel type porosity, this final honing must be controlled for other reasons; i.e., to give good clean cutting of the plateau surfaces, and avoidance of undue tearing up or breaking up of the porosity pattern which results if honing speeds, pressures and abrasives are too severe. The surface finish on the individual plateau should be very smooth.

A very hard, fast cutting abrasive is recommended, held in a soft or weak bonding material. An aluminum oxide or silicon carbide type abrasive, held in a resinoid bond, such as Bakelite, meets these conditions. Where only a single stone is to be used, a 180 or 240 grit abrasive is generally satisfactory for pitted or pin-point type porosity, and a 400 grit for channel and mechanical types; where two stones are used, a rough cut is taken on a 280 or 320 grit stone, and a finish cut with a 500 or 600 grit stone.

While honing is generally preferred where cylinders are to be finished, grinding or polishing can also be used. Lapping or sanding is commonly used on piston rings.

The honing or grinding operation leaves dirt and detritus, consisting of particles of honing stone and grit and abraded pieces of chromium, in the pores of the chromium plate. In many applications this is very harmful if not thoroughly removed. Ordinary cleaning or degreasing methods are ineffective for complete removal and, where thorough cleaning of porosity is important, it is accomplished by blasting the chromium surface under high pressure using organic solvent or alkali cleaner or other washing mediums. Cleaning of the porosity would seem advisable in any application, but in some it is imperative for successful functioning of the porous chromium deposit.

EQUIPMENT

As indicated in the above, from an operating standpoint the facilities required for porous chromium plating are much the same as for good hard chromium plating operations. No special solutions, tanks or electrical equipment is required beyond that found in a well equipped chromium plating shop.

The only special equipment, if the job is to be done completely by the plater, is the honing, grinding or lapping facilities, where ordinary hand stoning or wheel polishing is not suitable. Having the complete job done by the plater is generally recommended because of dependency of successful results on the close interrelation

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and coordination of the honing or finishing operations with the other two principal control factors, the plating and etching.

There should also be available optical inspection devices for examination and checking of porosity under a magnification ranging from about 5X to 50X. Where mechanical porosity is to be produced there would also be needed means of cutting or indenting the basis metal with engraving tools or etching facilities, or grit-blasting equipment.

In the plating operations proper, some shops might have to supplement existing current control facilities with additional equipment such as tank rheostats or independently controlled generators or rectifiers. Similarly, with bath temperature being such a critical factor, close automatic temperature control is a necessity; and with bath composition (ratio) so important, good practice requires analytical laboratory facilities close at hand.

SUMMARY

In summarizing, the following should be noted. Porous chromium plating involves more than producing merely a deposit having pores or crevices. It includes consideration of bearing surface design principles and requirements, and each job calls for careful analysis, execution and testing. With requirements known, however, porous chromium plating is not a difficult procedure from a process standpoint, but it is exacting in the same way that a precision hard chromium plating job is exacting. The type and degree of porosity are important; and porous chromium plate may be produced by both mechanical and chemical methods. In the latter, the porosity is dependent on three principal factors: the plating, the etching and the honing; all are interrelated in the final result. Finally, the plating operations proper for porous chromium are done in chromic acid solutions with which the plater is familiar, and require no special cleaning or racking methods; but conditions of operations do have to be closely controlled, all of which is accomplished with the usual and standard type of plating equipment and facilities.

ACID COPPER PLATING

By Dr. A. Kenneth Graham

Graham, Crowley and Associates, Inc.

Acid copper baths are used chiefly in electrotyping, electroforming and for immersion copper coatings. With the present tendency toward substantially heavier copper deposits as an undercoat for nickel and chromium, it is believed that the acid copper bath will be used to a greater extent in the coming year. In such applications the acid copper deposit will follow a substantial deposit from one of the cyanide baths. A satisfactory composition for general use is as follows:

Solution		Conditions	
Copper sulphate (crystals)	27 oz./gal.	Temperature	75 to 120°F.
Sulphuric acid	6.5 "	Current density	15 to 40 amp./ft.^{2}
		Voltage	0.75 to 2.0 volts
		Anodes	Rolled annealed copper

More rapid deposition is possible if air agitation is used. In such cases, the bath concentration is usually increased, the bath is warmed, and current densities of 75 to 150 amp./ft.^{2} are employed. The anode current density must not be excessive otherwise excessive polarization will occur.

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CYANIDE COPPER PLATING

By Dr. A. Kenneth Graham

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CONVENTIONAL COPPER CYANIDE BATH

Average Composition		Conditions	
Copper cyanide	3.0 oz./gal.	Temperature	75-100°F.
Sodium cyanide	4.5 "	Current Density	2.5-15 amp./ft.^{2}
Sodium carbonate	2.0 "	Voltage	1.5-2.0 volts
		Metallic copper	2.1 oz./gal.
		Free cyanide (average)	1.2 oz./gal.
		Anodes—Rolled annealed copper	

This bath may be used for still or barrel plating. Brighter deposits are obtained as the free cyanide is increased but the cathode efficiency is decreased under these conditions. Hypo (sodium thiosulphate) up to 0.25 oz./gal. is sometimes recommended to improve the brightness of the deposit. Smaller amounts are to be preferred and over-heating should be avoided as the hypo decomposes to sulphide and may cause roughness. Lead carbonate dissolved in caustic soda is also added as a brightening agent, but the very small amount required is not easy to control. An excess causes serious difficulty due to irregular results and brittleness. Hypo or lead should not be used if the deposit is to be oxidized. For plating cast or malleable iron, a lower free cyanide is recommended.

ROCHELLE SALT COPPER CYANIDE BATH

	Average Composition	Approx. Limits
Copper cyanide	3.5 oz./gal.	3-6 oz./gal.
Sodium cyanide	4.6 "	4-7 "
Rochelle salt	4.0 "	3-8 "
Sodium carbonate	4.0 "	2-8 "
pH	12.6 "	12.2-12.8 "

Conditions

Metallic copper	2.5 oz./gal.
Free cyanide	0.75 "
Temperature	140-160°F.
Current density	20-60 amp./ft.^{2}
Voltage	2-3 volts
Cathode efficiency	40-70%
Anodes	Rolled annealed copper
Anode area	2 times cathode area
Anode efficiency	50-70%

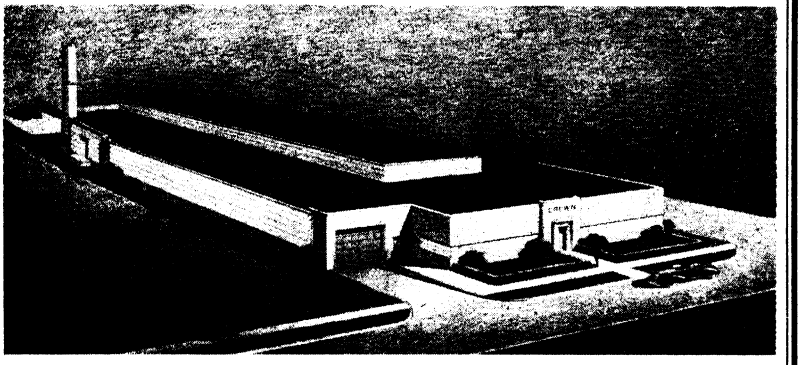
Time to deposit 0.0001 inch at 140°F.—
at 20 amp./ft.^{2} approximately 3.6 minutes
at 60 amp./ft.^{2} approximately 2.2 minutes

The conditions given cover the range within which bright deposits may be obtained. The higher the temperature, the higher the efficiencies. Agitation has little effect upon the cathode efficiency, but shifts the current density range within

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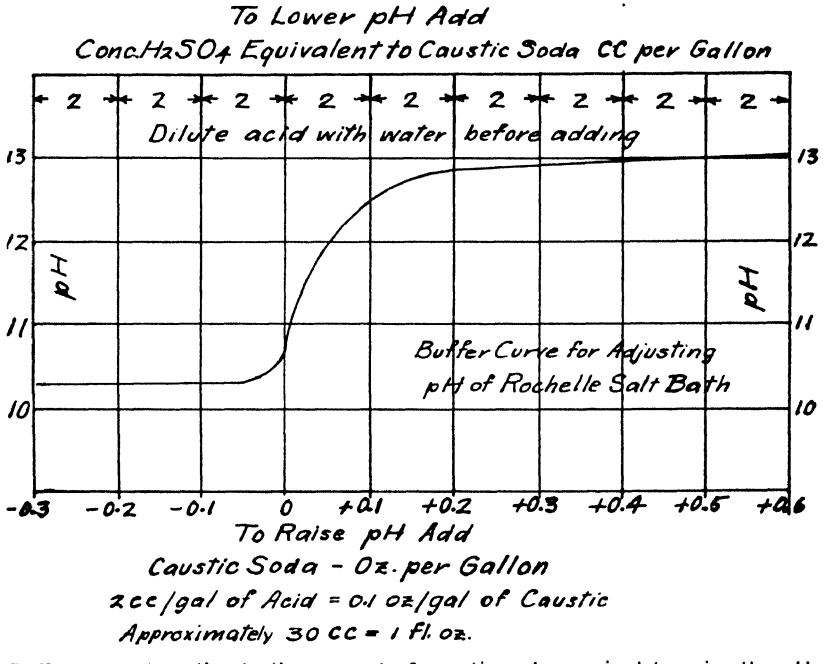
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which bright deposits are obtained. The anode efficiency is improved by agitation.

The free cyanide and pH must be controlled within close limits if uniform results are to be obtained. The pH can readily be raised by small additions of caustic soda. An excess should be avoided since it is necessary to add acid to lower the pH, and this offers complications due to the liberation of hydrocyanic acid gas which is poisonous. The buffer curve, Fig. 1, may be used to estimate the amount of caustic required to raise the pH, thus avoiding an excess. Should it become necessary to lower the pH, one to one sulphuric acid, tartaric acid or bicarbonate of soda may be used. Frequent additions of tartaric acid for this purpose will raise the Rochelle salt concentration to an unnecessarily high value. Bicarbonate should only be used if the extra carbonate will not prove excessive or where a change of pH is necessary without the liberation of excessive fumes. In such cases, the bicarbonate should be previously dissolved in water and thoroughly and rapidly mixed with the copper bath. This procedure should also be followed where either acid is employed.



Buffer curve to estimate the amount of caustic soda required to raise the pH

The carbonates should not be allowed to rise much above 8 oz./gal. otherwise the anode efficiency may fall rapidly at high current densities. On the other hand, the carbonates should not be removed below 2 oz./gal. (since iron will be attacked). One method of removing carbonates is by chilling the bath and separating the insoluble carbonate of soda. Another method is by the addition of gypsum to precipitate the calcium carbonate. The use of tartaric acid to control the carbonate concentration in a Rochelle salt bath has been suggested. If crys

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tals are added directly to the bath this method is satisfactory, but an excess of tartrates should be avoided.

It is recommended that at least 5% of the anode area be of iron in order to avoid excessive anode polarization at high anode current densities. The ratio of soluble to insoluble anode area can be varied to control the metal concentration of the bath. To avoid excess attack on the iron and formation of ferrocyanide, carbonates must be present in the solution and the pH must not be allowed to fall much below 12.0.

The conditions for bright deposits depend upon an adjustment of the temperature, current density and agitation. It should be noted that too violent agitation may be more harmful than none at all. The lower the copper concentration, the brighter the deposits, but the efficiency also decreases as the copper is reduced. Hypo and lead have both been used as brighteners, but their control is difficult and the former decomposes rapidly at the high temperatures employed. A high pH, i.e., 12.8, also favors brightness.

Iron in a Rochelle salt bath in excess of 0.75 g./l. has been shown to seriously reduce the anode efficiency and can not be removed. Other impurities such as zinc, lead, etc. affect the deposit. Electrolyzing the bath at low temperature and low current density (2 amp./ft.^{2}) is an effective method of restoring such a bath.

The deposits from a Rochelle salt bath can be readily oxidized. Less brushing is required and a brighter finish is obtained.

Barrel plating with a Rochelle salt bath is possible. Unless the volume of the bath and the anode area are adequate, difficulty is experienced in controlling the bath. Temperature control is also very important in such cases because high temperatures cause more rapid decomposition and carbonate formation. At high anode current densities, the latter may cause serious loss in anode efficiency.

Steel tanks may be used with cyanide copper baths although rubber-lined tanks are preferred. Automatic temperature regulation and chemical control are essential for consistent results.

Bright processes for rapid deposition of copper are available. For details regarding these, *Metal Finishing* should be consulted.

IMMERSION COPPER SOLUTION

A satisfactory bath for depositing copper by immersion on iron or steel articles is as follows:

Copper sulphate (crystals)	1 oz./gal.
Sulphuric acid	0.5 "

Best results are obtained if the temperature is not allowed to rise. All articles should be cleaned thoroughly before coating.

GOLD PLATING

By Joseph B. Kushner

Metal Finishing Consultant

In novelty and costume jewelry work the percentage of gold present in the plate is of little consequence; the color and appearance of the deposit are para-

mount. Thus a plater will speak of a "14 karat plate" when he means the color of the gold deposit is that of massive 14 karat gold. The actual amount of gold in such deposits usually runs between 19 and 22 karat. There are almost as many names for certain of the colored gold finishes as there are platers; the field has never been standardized. However, as formulated below, the colored golds can be grouped in four main divisions: yellows, whites, greens and pinks.

YELLOW GOLD

1. Potassium gold cyanide ½ oz./gal.
 Potassium carbonate 1 "
 Potassium cyanide ½ "
 Temperature 120-160°F.
 Current Density 5-15 amp./sq. ft. (2-6 volts)
 Anodes — Stainless steel, Nichrome, carbon or combination of any of above with gold.

2. Potassium gold cyanide ½ oz./gal.
 Potassium carbonate 1½ "
 Di-potassium phosphate 1½ "
 Potassium cyanide 1 "
 Temperature 140-160°F.
 Current Density 10-40 amp./sq. ft. (5-12 volts)
 Anodes As above
 This solution is used for gold flashing.

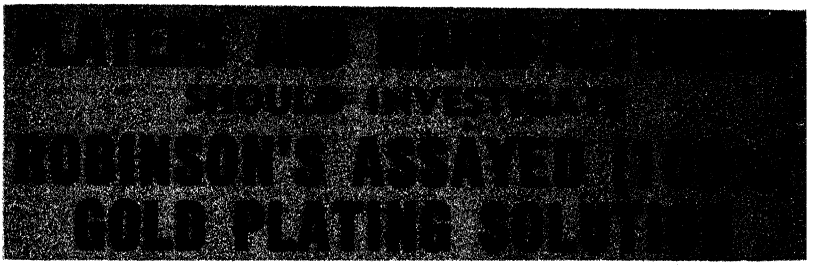
3. Potassium gold cyanide ½ oz./gal.
 Nickel formate saturated solution
 Temperature 80-90°F.
 Current density 1.8-2.2 amp./sq. ft.
 Anodes as above
 Keep solution saturated with nickel formate by having undissolved crystals at bottom of tank. The deposit from this solution is a pale yellow.*

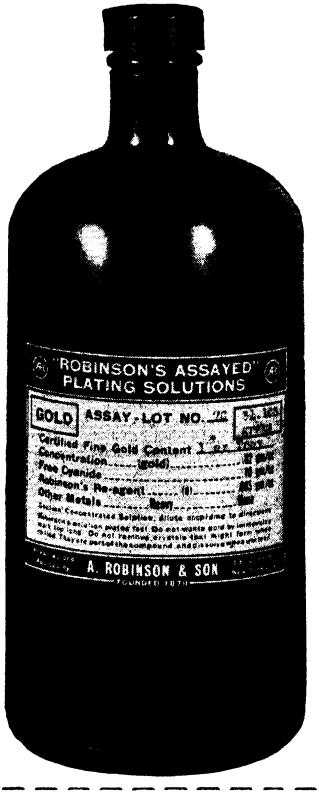
*U. S. Pat. #1,924,439

WHITE GOLD

1 Potassium gold cyanide ½ oz./gal.
 Nickel as double cyanide ¾ "
 Free cyanide 0.1 "
 Temperature 120-130°F.
 Current Density 40-200 amp./sq. ft.
 Anodes Stainless steel, pure nickel
 Deposits containing 15-20% Nickel can be obtained with this bath. A 15% nickel content gives a good white deposit.

2. Potassium gold cyanide 1 oz./gal.
 Potassium cyanide 2 "
 Potassium hydroxide 3 "
 Potassium stannate 11½ "
 Temperature 140-160°F.
 Current density 20-40 amp./sq. ft.
 Anodes Stainless steel





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They feature

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In calculating comparative costs, remember that

A Troy ounce contains 31.103 grams
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See Pages 70 - 267

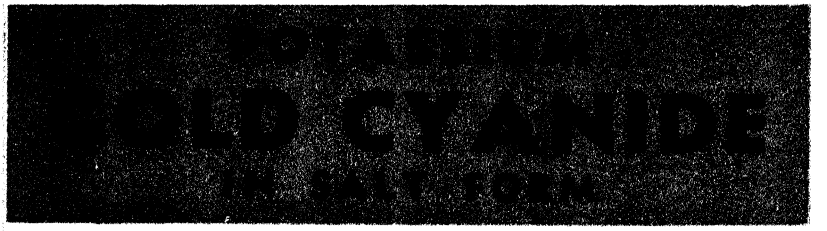
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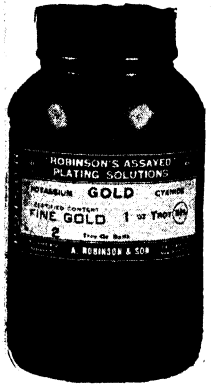
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- **Potassium Gold Cyanide** ☐
- **Rhodium Plating Solution** ☐

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- **Rhodium Plating Solution** ☐

NAME

STREET **CITY**

POSITION

Deposits with tin content below 30% show a slight yellow cast; greyness of alloys above this percentage can be removed by buffing.

GREEN GOLD

1. Sodium gold cyanide ½ oz./gal.
 Sodium silver cyanide 0.2 "
 Free cyanide ½ "
 Sodium carbonate ¾ "
 Trisodium phosphate 2¾ "
 Potassium sulfite ¾ "
 Temperature 120°F.
 Current density 10-20 amp./sq. ft.

2. Potassium gold cyanide ½ oz./gal.
 Cadmium oxide ¼ "
 Potassium hydroxide ¼ "
 Potassium cyanide 2.0 "
 Sodium bisulfite ¼ "
 Temperature 160°F.
 Current Density 10-30 amp./sq. ft.
 Anodes Stainless steel

The addition of small amounts of soluble arsenic and/or lead compounds to the green formulations given above darken the green shades; excessive admixture of the same produces smutted deposits.

PINK GOLD

1. Potassium gold cyanide ½ oz./gal.
 Copper cyanide ¼ "
 Potassium cyanide 3 "
 Temperature 120°F.
 Current density 10-15 amp./sq. ft. (3 volts)
 Anodes Stainless steel

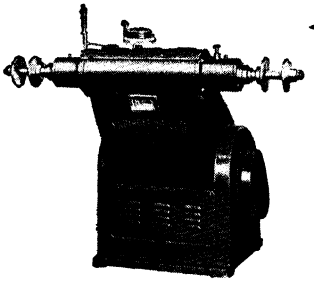
2. Sodium gold cyanide ½ oz./gal.
 Copper cyanide 1¾ "
 Sodium diphosphate 1¾ "
 Potassium cyanide 2½ "
 Potassium ferrocyanide ½ "
 Temperature 110-120°F.
 Voltage 3
 Anodes Stainless steel

ANTIQUE GOLDS

There are several methods for producing antique finishes. One most commonly used in the industry is to first put on a green smut finish from one of the green baths to which arsenic, lead or antimony has been added as described above. The smut is then relieved by any one of the common methods (bicarbonate of soda rub, short time tubbing, etc.) and the work is given a flash deposit in a yellow gold bath.

A method the writer found of value in producing certain desirable antique shades is the following:

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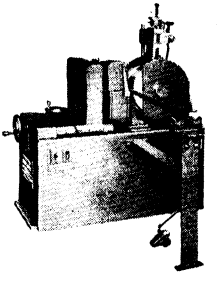


VARIABLE SPEEDS

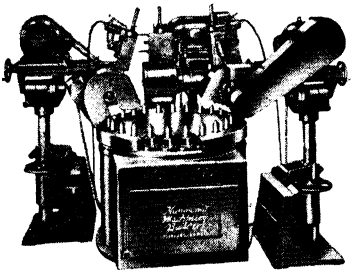
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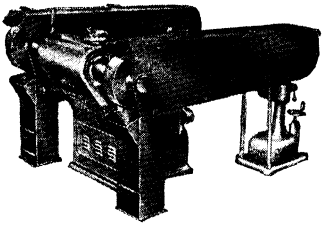


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1. Deposit a light flash of copper on the work.

2. Dip in a sulphide bath until copper plate is discolored.

3. Electrolyze as cathode for a few seconds in a mild alkaline solution such as 3 oz./gal. sodium carbonate. This reduces the copper sulphide, forming a heavy black smut on the work. Relieve as described previously and then flash in yellow gold.

LILAC GOLD*

Gold as fulminate	¼ oz./gal.
Potassium cyanide	2 "
Potassium zinc cyanide	6¾ "
Temperature	160°F.
Current density	900 amp./sq. ft.

*U. S. Pat. #1,905,106

At a zinc content of 30% the lilac color develops. It is important that the work is not agitated as agitation prevents the formation of the lilac or lavender color in the deposit.

The general principles to remember when plating out other metals with gold are:

1. Increasing current density increases base metal content of the deposit (all other conditions being the same).

2. Raising the temperature increases the gold content of the deposit (all other conditions being the same).

3. Agitation increases the gold content of the deposit (all other conditions being the same).

INDIUM PLATING

By M. A. Whitehead

American Smelting & Refining Co., Barber, N. J.

Indium is a soft, white, relatively tarnish-resistant metal which melts at 311°F. It has had wide acceptance for plating parts used in internal combustion engines, and for the development of surfaces capable of resistance to wear and corrosion, not only from acids in lubricating oils but also salt spray corrosion. For decorative purposes, it is possible to obtain a beautiful white finish, resembling silver, on steel by first coating the steel with a flash of non-ferrous metal, such as copper, brass or silver, and then plating and diffusing a light indium deposit. The diffusion is accomplished by heating at a low temperature, providing resistance to corrosion and wear and adding to the tensile strength of the alloys.

The most commonly used solution is the cyanide bath, but sulfate baths are gaining popularity rapidly.

SOLUTIONS

Cyanide Bath:[1]

Indium (as chloride)	4 tr. oz./gal.
Dextrose	2 av. "
Sodium cyanide	12 " "

SARCO LSI No. 87 CONTROLS





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SARCO SAVES STEAM

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The bath is prepared by adding the required amount of water to concentrated indium chloride solution in which form the indium is usually sold. Sodium cyanide is then added. A dark brown decomposition product forms during operation and is filtered off and processed for recovery of indium values. Insoluble anodes such as platinum, stainless steel or carbon are employed, the solution being replenished by additions of concentrated indium chloride solution. Operation is at room temperature and 10-150 amp./sq. ft., in glass, rubber lined or steel tanks.

High pH Cyanide Bath:

A high pH solution, which is more stable than the cyanide bath since the metal does not precipitate as the solution ages, has been developed by Mohler.[4] Indium chloride may be used to provide the metal content in the following:

Indium	15-30 g./L.
Potassium cyanide	140-160 "
Potassium hydroxide	30-40 "
Dextrose	20-30 "
Temperature	Room temp.
Current density	15-30 amp./sq. ft.
Anodes	Steel

Sulfate Bath:[2]

Indium (as sulfate)	2.4 tr. oz./gal. (minimum)
Sodium sulfate	1.3 av. "
pH	2.0-2.5

The bath is prepared by adding the required amount of water to a concentrated solution in which form the indium sulfate is usually available. The pH is adjusted by sulfuric acid or sodium hydroxide, as required. Since the anode efficiency is about 100% and the cathode efficiency will vary between 70 and 80%, combinations of indium anodes and insoluble anodes such as platinum or graphite are used. Throwing power is rather poor but the solution is simple to maintain. Operation is at room temperature and 20 amp./sq. ft., in glass, rubber lined, lead lined, mastic lined and ceramic tanks.

Fluoborate Bath:[3]

Indium fluoborate	31.5 oz./gal.
Boric acid	2.9-4.0 "
Ammonium fluoborate	5.3-6.7 "
pH (colorimetric)	1.0

This bath is operated at 70-90°F. with a current density of 50-100 amp./sq. ft. The pH is controlled by additions of 42% fluoboric acid solution. A cathode efficiency of approximately 75% is obtained under these conditions. The indium may be purchased as a 50% concentrate and is diluted to a metal content of 8.8 troy oz./gal. for use. Indium anodes will result in an increase of the metal content. It is therefore advisable to use carbon or platinum anodes with sufficient indium anodes to maintain the metal content constant.

1. U. S. Pats. 1,965,251 and 1,935,630.
2. H. B. Linford. Trans. Electrochem. Soc., 79, (1941).
3. H. Narcus. Metal Finishing, 43, (June 1945).
4. J. B. Mohler. Metal Finishing, 43, (Feb. 1945).

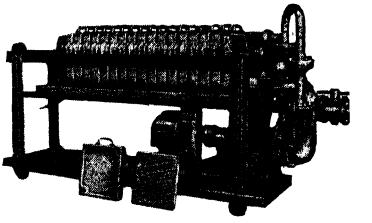
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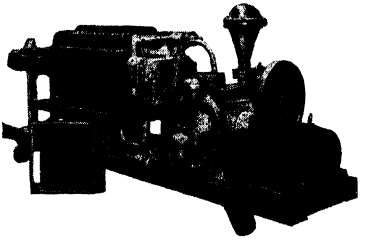
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Portable filter with lead filter plates and frames for high filtration rate and large holding capacity of solids, filter aids and carbon; equipped with alloy centrifugal pump.



Portable filter with rubber covered filter plates and frames and rubber covered Shriver Diaphragm Pump; capacity up to 2000 G.P.H.



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Write for Bulletin 108

T. SHRIVER & COMPANY

814 HAMILTON STREET HARRISON, N. J.

IRON PLATING

By C. T. Thomas[1] and V. A. Lamb[2]

(1) Foreman, Electrolytic Section, Bureau of Engraving and Printing, Washington, D. C.
(2) Chemist, National Bureau of Standards, Washington, D. C.

GENERAL

Iron plating has limited but increasing uses, which are likely to expand further as a result of the current shortages of many metals. Electroforming is the principal application. It is also used for building up worn or undersized machine parts, although nickel and chromium are generally preferred. After many attempts, electrorefining of iron has not been found economical. Under normal conditions, iron has been superseded by nickel and chromium as a hard facing deposit on softer metals.

Iron tubes have been produced in quantity by electroforming. Nickel-faced iron plates, electrolytically reproduced from engraved intaglio designs, are used in printing government bonds and currency. Experiments have been made recently on the substitution of electrodeposited iron for nickel and copper in letterpress printing plates. Increasing use has been made of iron plating for the reproduction of molds for various purposes.

Where it can be used, the low cost of iron gives it a great economic advantage over most other metals. Commercial high-purity iron suitable for anodes is available for 3c to 4c per pound. Soft, ductile adherent deposits up to a half-inch or more in thickness can readily be produced. These machine well and can be hardened by carburizing or nitriding. They can be readily welded by the usual methods. Relatively hard deposits can also be made. These are more brittle, and in some cases have a tendency to crack, especially with increasing thickness. They can be rendered soft and ductile by heat treatment. The high magnetic properties of electrolytic iron may also be useful.

PLATING SOLUTIONS

Chloride Baths

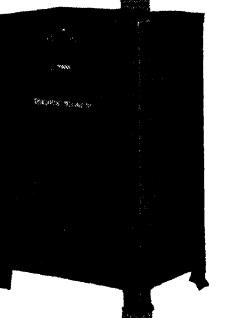
For the production of soft and ductile metal, a concentrated solution of ferrous chloride makes an excellent bath. Calcium chloride is usually also present, and, although not essential to good results, its use is advised. A recommended formula is:

1.	Ferrous chloride	50 oz./gal.
	Calcium chloride	25 "

These concentrations may be varied widely, but the ferric iron content must be kept low. This bath must be used hot, at temperatures of 85°C. (185°F.) or higher. For a current density of 60 amp./ft.^{2}, which is generally used, a temperature of 90°C. (194°F.) is recommended. It has been shown that metal of excellent quality can be obtained at current densities as high as 300 amp./ft.^{2} under carefully controlled conditions. Finally, the bath must be maintained in a slightly acid condition, which usually requires periodic additions of hydrochloric acid. An acidity of approximately 0.01 *N* HCl (0.365 g. HCl/liter) is used with average current densities. This can be controlled most conveniently by means of

Flexible power

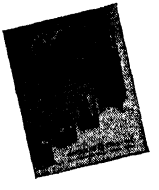
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Output voltage of a single unit, or of a multiple-unit group, is accurately controlled on the a-c input side by G-E rectifier control. By substituting this "on load" control for tank rheostats, important power savings are realized. Control can be automatic or manual. With G-E automatic control, voltage is maintained within plus or minus two per cent, from no load to full load.



There are many G-E plating and anodizing rectifier and control combinations of different ratings built to meet your exacting needs. For illustrated booklet and full information, write General Electric Company, Appliance and Merchandise Department, Section A561-62, Bridgeport, Connecticut.

GENERAL ELECTRIC

pH measurements with a glass electrode. A pH range of 0.6 to 1.0 is satisfactory. In a reduced solution the acidity can also be measured by titration with dilute alkali, using methyl orange as indicator.

A new bath or one that has stood idle for some time will usually contain an appreciable amount of ferric chloride. This will be evident from the color of the solution, which should be a clear green in the reduced or ferrous condition. The color takes on a yellow tinge which deepens to a dark brown with increasing concentration of ferric or oxidized iron. Before use, the bath must be given a reducing treatment by adding an excess of hydrochloric acid and allowing it to stand overnight or longer in contact with scrap iron or anodes. Warming hastens the action. Operating the bath with dummy cathodes also reduces the content of ferric iron.

The rate of oxidation of the bath is quite low. This is especially true if the anodes are removed and a slight excess of acid is maintained when the bath is not in use. Small blocks of rubber floated on the surface have been found by one author to reduce this tendency, and also to conserve heat and reduce evaporation and spray. The bath is not sensitive to the inorganic impurities usually encountered but most organic substances are definitely harmful. With a little experience, control of this type of bath will be found easy and simple, especially if it is used regularly. Air agitation is likely to cause brittle deposits.

Another hot chloride solution which was recently described[1] has the following composition, operating ranges, and operating characteristics:

2.	Ferrous chloride	26 to 65 oz./gal.
	Manganese chloride	0.39 to 0.65 "
	Gardinol wetting agent	0.13 "
	Temperature	160 to 220°F.
	pH	1.5 to 2.5
	Current density	50 to 300 amp./sq. ft.
	Cathode current efficiency	95%

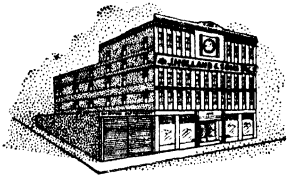
The lowest temperature at which ductile deposits can be obtained from this bath is about 160°F., and at this temperature the maximum current density is about 50 amp./sq. ft. It is claimed that the addition of the manganese chloride widens the temperature and pH ranges over which ductile deposits may be obtained, and results in deposits having a smaller, more uniform grain size.

Because of the strongly corrosive nature of these baths at high temperature, tanks and fittings in contact with the solution must be made of acid-resistant materials, or be given an acid-proof coating. Tanks lined with rubber, with acid-proof brick, or with both of these, are recommended as containers. Auxiliary equipment lined with glass or rubber, or made of materials like high-silicon iron or ceramic may be used with proper precautions.

Even in Armco iron anodes, inclusions are present which do not dissolve, and are carried over to the cathode or plating surface, causing rough deposits of poor quality. Bagging the anodes with pure blue African asbestos cloth filters out these particles. Woven glass cloth has been used successfully for this purpose, and the use of porous rubber is being investigated.

The chloride baths described above must be operated at a high temperature. The following chloride bath has been found to produce smooth, strain-free deposits at room temperatures:

3.	Ferrous chloride	32 oz./gal.
	Potassium chloride	24 "

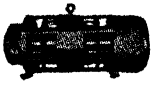


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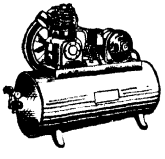
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The concentrations may be varied widely. The iron deposited from this bath at room temperature is brittle, but its ductility increases with a rise in temperature of deposition. For deposition on a wax electrotype mold, the upper temperature limit is about 35°C. (95°F.). Satisfactory deposits have been obtained at temperatures as low as 20°C. (68°F.). Current densities up to 50 amp./ft.^{2} can be used at 35°C. (95°F.). The pH of the bath should be maintained in the range of 5.0 to 5.5 by periodic, slow additions of potassium hydroxide solution. The pH may be measured with a glass electrode or colorimetrically with bromocresol purple. If the latter method is used, a "salt error" correction of approximately 0.5 pH is involved. Thus, a "true pH" of 5.0 is equivalent to a "colorimetric pH" of 5.5. Since oxidation by the air will rapidly change the pH of a sample, a freshly withdrawn and unfiltered sample should be tested. This bath oxidizes slowly, which results in the formation of a precipitate of ferric oxide. The precipitate does no harm in electrotyping applications, and anode bags are usually unnecessary.

The maintenance of the bath is similar to that of the hot ferrous chloride-calcium chloride bath described above. If low temperatures are employed, a bituminous tank lining may be satisfactory.

Sulfate Baths

Sulfate baths have been used for a long time, and still have a limited use. Because this type of bath can be used at lower temperatures than the ferrous chloride-calcium chloride baths, is less corrosive, is less subject to air oxidation, and tends to give harder deposits, it is well adapted for use in building up machine parts. This is its principal role. A simple and much used bath of this kind is a concentrated solution of ferrous ammonium sulfate. A concentration of 50 oz./gal. of the salt allows the use of a maximum current density, but concentrations less than this may be used. The content of ferric iron must be kept low, which may be done with a reducing treatment similar to that used for the chloride baths, except that sulfuric acid is added in place of hydrochloric acid.

At room temperature a current density of 20 amp./ft.^{2} is used. Higher temperatures allow higher current densities. At 60°C. (140°F.) 60 amp./ft.^{2} may be used. The bath may be operated in either a slightly acid or nearly neutral condition. In the acid range, acidities may vary from 0.005 *N* H_2SO_4 (0.245 g. H_2SO_4/liter) at low temperatures and current densities to 0.02 *N* H_2SO_4 (0.98 g. H_2SO_4/liter) for rapid deposition.

Good deposits are also obtained from this bath when operated in the pH range of 5.0 to 5.5. The method of pH measurement is the same as that described for the chloride bath. The pH may be controlled by making periodic additions of ammonia. Addition of a suspension of fresh ferrous carbonate is also reported to be a good method of maintaining the pH in this range. In this case, powdered charcoal is sometimes added to the bath to reduce pitting. Gentle mechanical agitation to keep the ferrous carbonate and charcoal in suspension is desirable.

Iron Plating on Stereotypes and Electrotypes

Any of the above described baths may be used for plating on stereotypes and electrotypes. The ferrous chloride-potassium chloride bath and the sulfate bath have been used with success on an experimental scale. For this purpose, the pH range of 5.0 to 5.5 is recommended for the sulfate bath. Adequate cleaning of the printing plate surface prior to plating is essential.

Iron-plated stereotypes have been run for over 1,000,000 impressions with no apparent wear, and iron-plated copper electrotypes for over 4,000,000 impressions.

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Deposition on Wax and Lead Molds

The sulfate bath described above cannot be used to deposit iron directly on wax and lead molds. The ferrous chloride-potassium chloride bath is recommended for this application.

A modified sulfate bath described recently[2] is also recommended for application to wax mold electrotypes. The solution has the following composition and operating conditions:

Ferrous sulfate	33 oz./gal.
Ferrous chloride	5.6 "
Ammonium chloride	2.7 "
Temperature	100 to 105°F.
pH	4.5 to 6
Current density	50 to 100 amp./sq. ft.

The bath is also recommended for plating on stereotypes. In this application a slightly lower temperature and pH are used, e.g., 90°F., and 3.5 to 5.5, respectively.

When either of the above baths is used for depositing iron on wax molds, the graphited wax molds (called "cases") must first be coated with copper by treatment with copper sulphate and iron fillings or powder, a process known to electrotypers as "oxidizing" the cases. Best results are obtained if this process is applied twice, or if after one oxidizing treatment the cases are "flashed" in an acid copper bath for 1 minute, after which iron is deposited.

Silvering of the mold by chemical reduction methods with silver spray equipment has also been used successfully in preparing the molds to receive a deposit. On lead mold electrotypes the same treatment is used as for nickel deposition.

On flat printing plates about 0.004" of iron and 0.002" of copper may be used, and on plates to be curved, about 0.002" of iron and 0.004" of copper. All the nickel and part of the copper commonly used in electrotyping may thereby be saved.

1. W. B. Stoddard, Jr., Trans. Electrochem. Soc. 84, preprint 5 (1943).
2. R. M. Schaffert and Bruce W. Gonzer, Trans. Electrochem. Soc., 84, preprint 25 (1943).

LEAD PLATING

By F. C. Mathers

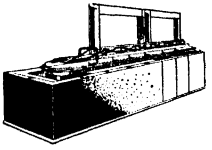
Indiana University, Bloomington, Indiana

GENERAL

Lead makes a protective coating which is superior to that of any other metal against acid fumes and corrosive liquids. This is especially true as regards resistance to corrosion by sulfuric or hydrofluoric acid. Lead differs from zinc, as a protective covering, in that zinc continues to protect even when the coating is broken, whereas lead, if broken or pitted, allows the basis iron to rust or corrode. This is due to the fact that lead is cathodic to iron just as is copper and most other metals. Recent tests, however, seem to indicate that rust which may form at pin holes in lead deposits does not tend to spread under the coating as in the case of other cathodic coatings.

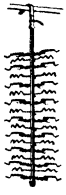
Freedom from pin holes rather than thickness or weight of lead per sq. ft. is

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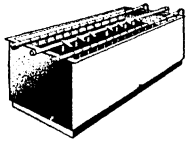
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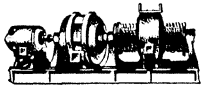


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ACID STRIPPING AND DESCALING AGENTS. Remove rust, corrosion and scale deposits . . . protect metal surfaces from harmful acid attack . . . control acid fuming. Can be used with safety on iron, steel, copper, brass, bronze, nickel and their principal alloys. Easy to work with.



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the criterion for the protective value of lead deposits. Deposits of 0.003" (0.075 mm.) will usually be impervious, although thicknesses varying from 0.0008" to 0.12" (0.02-3 mm.) are mentioned as being used. Gray and Blum[1] recommend 0.005", but if resistance to heavy corrosion is necessary, 0.05" or more should be used. Lead is malleable and ductile; hence plated articles can generally be "worked" without causing breaks. It should also be mentioned that lead is cheaper than the other metals. Lead is too soft to resist wear or abrasion and is much too toxic for use in contact with food products. Imperviousness of the deposits is of no importance where the lead is used for anti-friction in bearings.[1] A thickness of 0.0001" will protect[11] steel in salt spray for 18-30 hours.

A review, complete to that date, on the electrodeposition of lead is found in Trans. Electrochem. Soc., **23**, 153 (1913). Detailed working directions and a good bibliography for the various lead baths are given in Metal Industry (N.Y.) [now Metal Finishing (N.Y.)] **13**, 184, (1915). The recent paper by Gray and Blum[1] gives an excellent discussion and description of lead plating practices with both the fluosilicate and the fluoborate bath.

Continued research has finally shown that almost any soluble lead salt will produce dense, smooth deposits, if the proper organic addition agents are used. Difficulties encountered in attempts to use certain lead salts are now known to have been caused by not using the right addition agents. Lead plating is not difficult. The limited solubility of the lead salts of the more common acids such as the sulfate, chloride, and fluoride, and the oxidizing property of nitrates, have made necessary the use of lead salts of uncommon acids.

LEAD FLUOSILICATE BATHS

The lead fluosilicate[1] bath is not recommended[11] for lead plating because the deposits are not as good as those from the fluoborate and, sometimes, a precipitation of silicic acid and lead fluoride causes trouble. On account of its lower cost, the fluosilicate bath is still used for lead refining.[5] The composition[3] of the bath is not critical, and solution, ready for use, can be purchased from lead refining companies. It is not recommended that any plater attempt to manufacture the bath—a rather difficult and uncertain process. If this bath is used, the plater should remember that plenty of glue—as much as 3 g./l. is important for good deposits—and also goulac, perhaps 3 g./l., is desirable. More of these addition agents must be added as needed. The use of 2.5 g. of glue for each 2 lb. of lead deposited is recommended.[4]

LEAD FLUOBORATE BATHS

The lead fluoborate bath[1, 9, 10, 11, 12, 13] is generally used for plating. Compared with the fluosilicate bath, it gives finer grained, denser deposits; it is less susceptible to decomposition, and it gives a satisfactory plate directly on iron or steel; but it is more expensive. Another advantage is that now a concentrated solution all ready for plating except for addition agents and the proper dilution can be purchased.[2] It is better[11, 13] to buy this bath ready for use, except, usually, for addition agents, than to attempt its manufacture in the plating room. It is also possible to buy[2] a concentrated solution of fluoboric acid from which the bath can be made by adding the lead carbonate and addition agents. The composition of the bath can be varied through wide limits without producing much change in the character of the deposits.

The fluoboric acid, as purchased, contains 42% acid and has a specific gravity of about 1.3. A bath from this acid is made up as follows:

	oz./gal.	g./l.	cc./l.
Fluoboric acid, 42%	42.5	320	246
Basic carbonate of lead, 2 $PbCO_3 \cdot Pb(OH)_2$	17.0	130	
Glue	0.03-0.6	0.2-5	

The final bath has a composition about as follows:

	oz./gal.	g./l.
Lead as fluoborate	14.	105
Free fluoboric acid	5.3	40
Glue	0.03-0.7	0.2-5

If the bath is made starting with hydrofluoric acid, the materials used are:

	oz./gal.	g./l.
Boric acid	14	105
Hydrofluoric acid, 48%	32	240
Basic lead carbonate, 2 $PbCO_3 \cdot Pb(OH)_2$	20	150
Glue	0.03-0.7	0.2-5

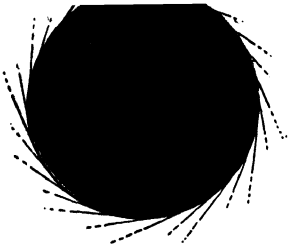
The lead fluoborate concentration[9] should be 16 oz./gal. (120 gms./l.) for obtaining thick deposits.

The boric acid is added slowly to the hydrofluoric acid, diluted with an equal volume of water, in a hard rubber or lead vessel. Much heat is developed, hence care is necessary, and too rapid mixing will result in the volatilization and loss of acid. After the solution is cool, the basic lead carbonate, "white lead," in the form of a thick paste with water, is added slowly with stirring. Much carbon dioxide will be evolved, and there is danger of the solution foaming over. After the insoluble matter has settled, the clear solution is siphoned off, or filtered, and diluted to the required volume. The bath is ready for use after addition of the glue.

The use of goulac, a residue from sulfite paper manufacture, equal in quantity to the glue used, improves the deposits. These addition agents produce a precipitate which can be removed by filtration or by settling. Some excess of boric acid reduces the tendency for decomposition of the fluoboric acid with precipitation of lead fluoride. An excess of free fluoboric acid reduces treeing from the corners of the cathodes and gives finer grained deposits.

It has been the experience in this laboratory that more glue than usually recommended makes the deposits better. There must always be a compromise on the quantity of addition agent because, although smoother deposits are obtained with larger quantities, the deposits become darker and more brittle as the addition agent is increased. It has been reported that the accumulations of glue residues in the baths finally become injurious. A higher concentration[8, 9] of glue than 0.02-0.033 oz./gal. (0.15-0.25 g./l.) is said to be detrimental by some platers.

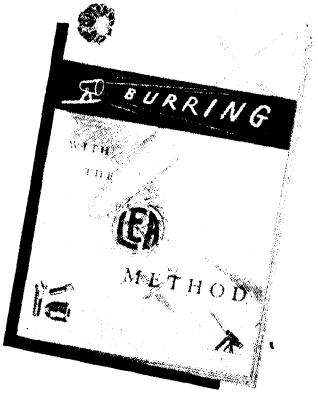
The current density that can be used depends upon how thick and how excellent a deposit is desired, the concentration of the solution, and the degree of agitation or stirring. Each plater must decide the current density that best suits his situation. However, as high as 80 amp./ft.^{2} (8.6 amp./dm.^{2}) can be used. This writer prefers the lower current densities and suggests 10 20 amp./ft.^{2} (1-2 amp./dm.^{2}) at room temperature. Lead deposited at 0.1 amp./sq. ft. has better wear resistance[8] than deposits made at higher current densities.



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Pure lead anodes, preferably from electrolytically refined lead, should be used. The current efficiency is about 100% and voltage is between 0.1 and 0.2. Cleaning of the articles to be plated by sandblasting is better than acid cleaning, and any acid cleaning should be very light. Glass vessels are not attacked very rapidly by this bath. The plater should remember, if he attempts to make the bath, that concentrated hydrofluoric acid, or its vapor from a hot solution, produces very painful burns on the skin. Fluoboric acid and fluoborates do not cause this trouble.

ALKALINE LEAD BATHS

A recommended composition[6] is:

	oz./gal.	g./l.
Lead acetate	10	75
Sodium hydroxide	27	200
Rosin	0.4-1.3	3-10

A portion of the bath should be warmed[7] with rosin, stirred well, cooled, filtered and added to the rest of the bath. Glue can be used but it is destroyed rapidly by the hot alkali and must be renewed every 2-3 days.

The higher the temperature, the better the deposit—194°F. (90°C.) being noticeably better than 176°F. (80°C.). A current density of 10-20 amp./ft.^{2} (1-2 amp./dm.^{2}) can be used, but the lower the current density, the better the deposit. The anodes do not dissolve very well; hence the anode current density should be kept below 10 amp./ft.^{2} (1 amp./dm.^{2}). These lead deposits do not give as good protection to iron as do those from acid solutions. The addition of sodium-potassium tartrate (6.6 oz./gal.), is recommended.[7]

1. Gray and Blum, Trans. Electrochem. Soc., 80, 645 (1941).
2. Many plating supply companies can furnish lead fluoborate solution.
3. Blum and others, Trans. Electrochem. Soc., 36, 248 (1919).
4. Reeve, Trans. Electrochem. Soc., 35, 389 (1919).
5. Betts, Electrochemical Ind., (now Chem. and Met. Eng.), 1, 407 (1903).
6. Mathers, Trans. Electrochem. Soc., 38, 121 (1920).
7. Chem. Abst. 36, 4031 (1942).
8. Wright, J. Electrodepositers' Tech. Soc., 20, 1 (1945).
9. Diggin, Metal Finishing, 41, 418 (1943).
10. Mullin, Monthly Rev., Am. Electroplaters' Soc., 31, 898 (1944).
11. Beall, Ibid., 31, 719 (1944).
12. Bray, Steel, 115, No. 15, 128 (1944).
13. Narcus, Metal Finishing, 43, 188 (1945).

NICKEL PLATING

By Derick S. Hartshorn, Jr.

The Enthone Co., New Haven, Conn.

DULL WHITE NICKEL SOLUTION

Single nickel salts	16 oz.	pH, colorimetric	5.4-6.0
Ammonium chloride	3 "	Anodes	99+%, rolled depolarized
Boric acid	4 "	Temp.	75-85°F.
Water to make	1 gal.	Current Density	5-15 amp./ft.^{2}
		Voltage	1½ to 2

Solution Limits:

For nickel plating at room temperature, double nickel salts are sometimes

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COPPER SULPHATE

NICKEL SULPHATE

COPPER OXIDE

used in place of part of the single nickel salts. However, it is easier to control the solution if the ammonium radical is added as NH_4Cl. Too great an excess of ammonium chloride or double nickel salts will result in precipitation of double nickel salts when the solution cools because the latter salts are only moderately soluble at room temperature (68°F.), the solubility being 13.9 oz./gal. The ammonium radical allows a higher current density to be used than when it is not present.

Operating Notes:

Pitting can be overcome by the use of hydrogen peroxide in concentrations of the order of 1 cc. of 3% peroxide (10 vol.) per gallon of solution. An excess may cause brittleness or poor throwing power, but this condition can be remedied by heating the solution to 160°F. for about an hour.

Brittleness of the deposit can be caused by impurities, too low solution temperature, too high current density and too high pH.

Dirt pitting can be overcome by bagging the anodes and filtering the solution.

A rapid rise in pH may be caused by a low boric acid content, low cathode efficiency (solution low in metal) or by the drag-in of alkali from cleaner or cyanide dip. A rapid drop in pH may be caused by a low boric acid content, low anode efficiency (low chloride content), or by the drag-in of acid from poor rinsing of the work after acid dipping.

Lowering of the pH can be done by adding hydrochloric acid (if the chloride is low) or by adding sulfuric acid. The best way to determine the amount of acid required, is to titrate a standardized dilute acid into a small sample of the solution until the desired pH is obtained, and then calculate the amount of acid required for the entire tank. The pH can be raised by stirring in nickel carbonate or hydroxide and then filtering and readjusting the pH with acid, if too high. Ammonia should only be added if the ammonium chloride content is low, and then an amount equivalent to 3 oz./gal. of NH_4Cl only, can be used. The chloride can then be added using nickel chloride.

BARREL NICKEL SOLUTION

Single nickel salts	20 oz.	Water to make	1 gal.
Ammonium chloride	4 "	Temp.	75-90°F.
Boric acid	4 "	pH, colorimetric	5.4-6.0

DIE CAST NICKEL

For plating on zinc and its alloys directly, the following solution is employed where heavy deposits are not required. The deposits are brittle and tend to crack when applied in greater thickness.

Single nickel salts	10-15 oz./gal.
Sodium sulfate, anhydrous	10-15 "
Ammonium chloride	2 "
Boric acid	3 "
Temperature	Room temp.
Current density	10-35 amp./ft.^{2}
Voltage	2-3 volts
pH, colorimetric	5.8-6.2

If crystal sodium sulfate is employed instead of the anhydrous form, double the amount.

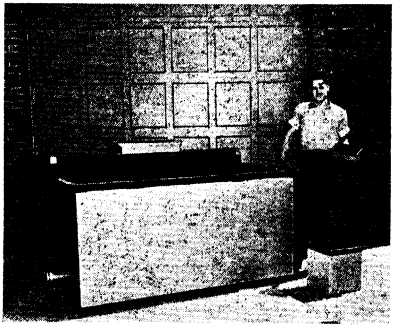
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WARM NICKEL (WATTS TYPE)

Single nickel salts	25-45 oz./gal.	Temp.	120-160°F.
Nickel chloride	6-9 "	Current density	15-50 amp./ft.^{2}
Boric acid	4-6 "	pH, colorimetric	5.2-5.8

Nickel solutions should be put preferably in rubber-lined tanks, as lead-lined and pitch-lined tanks may lead to contamination of the solution, particularly the latter if wetting agents are used in the solution.

If wetting agents are used to control pitting, brittleness of the coating should be watched both from the wetting agent and its decomposition products. Activated carbon treatment will usually remove most of the materials causing brittleness if they are of an organic nature.

Agitation of the work or solution will enable considerably higher current densities to be used, particularly if the work is agitated. Agitated solutions should be filtered continuously or regularly to remove solid matter that may cause roughness.

Rapid pH changes may be due to the causes outlined for the cold nickel solution described above.

SEMI-HARD NICKEL*

Nickel chloride	40 oz./gal.	Anodes	99+%
Boric acid	4 "	Temp.	140°F.
pH, electrometric	2.0	Current Density	20-100 amp./ft.^{2}
Hardness (Vickers)	230-260		

HARD NICKEL*

Single nickel salts	24 oz./gal.	Anodes	99+%
Ammonium chloride	3.3 "	Temp.	120-140°F.
Boric acid	4.0 "	Current Density	25-50 amp./ft.^{2}
pH, colorimetric	5.6-5.9		
Hardness (Vickers)	380-500		

Operating Notes:

Higher pH, higher current density and lower temperatures will result in harder deposits. Lower pH, lower current density and higher temperatures will result in softer deposits.

Hard and semi-hard nickel deposits are used where a deposit is required which is tougher and less liable to crack and peel than is chromium, yet where the extreme hardness of chromium is not essential. They are also used for corrosion and wear resistance.

REFERENCE

*International Nickel Co., Inc., Instruction Sheets, 1941.



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BLACK NICKEL PLATING

By John G. Poor

Van der Horst Corporation of America, Olean, N. Y.

GENERAL

Black nickel must be considered as a purely decorative finish and should not be directly applied to ferrous base metals as a corrosion resistant coating. The deposit cannot be considered to be metallic and does not exhibit appreciable ductility nor malleability which are properties common to most metals. Very thin deposits will withstand moderate bending tests and are sufficiently adherent to be of practical value. Heavy deposits can be readily obtained in warm solutions; however, they are liable to chip or flake off on bending or impact.

In finishing ferrous base metals, regular specification coatings of metals such as nickel, cadmium or zinc should first be deposited to meet corrosion resistance requirements. Black nickel may be deposited over copper, nickel, brass, cadmium and zinc or electrodeposits of these metals. Uniformly lustrous and smooth deposits are comparatively easy to obtain over cadmium and zinc and such coatings are quite adherent as shown by bending tests. Over regular white nickel coatings, the apparent adhesion is not quite as good and more careful control of operating conditions is required for uniform, smooth, black deposits. Deposited over nickel, the finish is more resistant to scratching and marring than when deposited over relatively soft metals.

EQUIPMENT

Rubber lined steel tanks are most suitable for black nickel plating. Tank capacities should be extra large relative to the volume of work to be plated so that the composition of the bath as a whole will change less rapidly during use and require fewer analyses and additions for its maintenance.

Temperature control is especially important. Suitable means for heating the solution and maintaining temperature within rather close limits is almost essential.

The current density range within which good deposits can be obtained is comparatively narrow so that adequate rheostats and meters are necessary. Separate current supply for black nickel plating is sometimes desirable where main bus bar voltages fluctuate because of the addition or withdrawal of heavy current loads at other plating tanks.

Plating racks and the contacts inside plating barrel cylinders should be stripped of their black nickel deposit at the end of each plating cycle so as to insure good electrical contact with new batches of work. Strong hydrochloric acid or an acid bright dip, such as is used for brass, will strip the deposit very readily.

Anodes

In solutions operated at room temperature and at relatively high pH values, gas carbon anodes seem to be the most satisfactory. Stainless steel anodes may also be used or a small proportion of nickel anodes may be used along with the insoluble anodes.

In warm solutions at lower pH values, regular 99+% nickel anodes are used with, or without, other insoluble anodes. Under the latter conditions, nickel anodes are less apt to become coated with difficultly soluble corrosion products and the volume of sludge accumulating in the bath is reduced.

Agitation

Agitation of the work or of the solution is not generally necessary except

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where baths of low concentration are operated at higher current densities. If agitation is used, great care should be taken to prevent aeration of the solution which invariably causes pitting of deposits.

SOLUTIONS

The solution composition given under formula No. 1 is especially recommended because a better knowledge of its operating range and characteristics has been obtained. Furthermore, the higher initial concentration of bath constituents permits a longer period of satisfactory operation before replenishment becomes necessary.

Formula No. 1

Single nickel salts	10 oz./gal.
Double nickel salts	6 "
Zinc sulfate, crystals	5 "
Sodium thiocyanate	2 "

Formula No. 2

Double nickel salts	8 oz./gal.
Zinc sulfate, crystals	1 "
Sodium thiocyanate	2 "

Formula No. 3

Double nickel salts	12 oz./gal.
Zinc sulfate, crystals	3 "
Sodium thiocyanate	0.5 "
Boric acid	2.0 "

It is recommended that bath constituents be maintained within 10% of the values given in the above formula. The concentration of zinc sulfate and sodium thiocyanate fall more rapidly below proper limits and must therefore be more frequently determined by analysis. If these deficiencies are not corrected by proper additions, deposits will tend to be grey. Boric acid may be added to formula No. 1 as a buffering agent; however, satisfactory deposits are then obtained at lower pH values and within more narrow ranges of temperature and current density.

Operating Conditions (all formulas)

Temperature	75-95°F.
pH	5.8-6.1 electrometric, quinhydrone
Current density	0.5-1.5 amp./ft.^{2}
Plating time	20-40 minutes

Under these conditions, and especially with insoluble anodes, the pH value will drop as the bath is operated. Ammonia or nickel carbonate is added to maintain the pH value within proper limits.

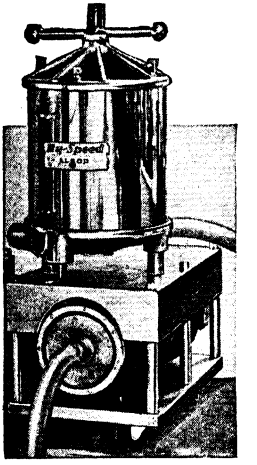
Operation at too low a temperature may result in rough or burned deposits and reduces the current density at which satisfactory black deposits can be obtained. Too high a temperature at a given current density results in the deposition of grey or of powdery grey coatings.

Deposits may be either soft or light in color if the pH value is too low, while basic zinc salts may be precipitated in objectionable amounts when the pH value is excessively high.

Operation at too high a current density at a given temperature will cause rough or burned deposits.

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Other Operating Conditions (Formula 1)

Satisfactory deposits can be obtained in considerably less plating time using the following operating conditions:

Temperature	120-130° F.
pH	5.6-5.9 electrometric, quinhydrone
Current density	5-20 amp./ft.^{2}

Under these conditions and with a large proportion of nickel anodes the pH value rises. Sulfuric acid is added to maintain the pH within proper limits.

GREY DEPOSITS

Very smooth, adherent, grey deposits can be obtained using solution formula No. 1 under the following conditions:

Temperature	120-130° F.
pH	5.6-5.9 electrometric, quinhydrone
Current density	0.5-2.5 amp./ft.^{2}

PLATING METALS OF THE PLATINUM GROUP

PALLADIUM, PLATINUM, RHODIUM

By Dr. K. Schumpelt

Baker & Co., Inc., Newark, N. J.

PALLADIUM

Both the old Pfanhauser formula as well as the Keitel and Zschiegner formula can be used for making palladium solutions. A palladium salt corresponding to the platinum diammino nitrite is used in the latter case. Good palladium deposits, however, can be obtained only at voltages below the gassing point; about 1.2-1.5 volts. The acid chloride bath of Wise and Vines is suitable for heavy deposits.

1. Diammino-nitrite Baths:

Palladium ammino nitrite (Dissolved in small amount of ammonia)	8 grams
Ammonium nitrate	100 "
Sodium nitrate	10 "
Water	1 Liter

If the solution is operated with a pH above 7 by the addition of ammonia, the deposits become smoky rather quickly, but heavier deposits can be built up by means of intermittent scratch brushing. If the pH is below 7 the deposits have a tendency to be brighter, but the danger of peeling increases.

2. Sodium Palladium Nitrite Bath:

Sodium palladium chloride	10 g./L.
Sodium nitrite	10 "
Sodium chloride	50 "

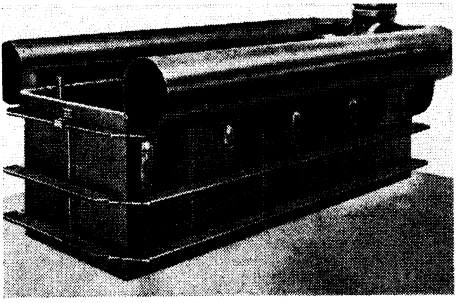
The solution is prepared by dissolving the palladium salt in about 500 cc. of water, after which the sodium nitrite is added. The sodium chloride is finally added and the solution diluted to 1 liter. The bath is operated at about 50° C. and 10 amp./sq. ft. requiring about 1.2 volts. Palladium anodes are used and the efficiencies of anode and cathode are about 100%. The pH should be maintained at

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4-5 by additions of hydrochloric acid or sodium carbonate, as required. The deposits stay bright for a long time, and if a slight cloudiness appears it can easily be buffed away.

3. Acid Chloride Bath:

Palladium (as chloride)	50 g./L.
Ammonium chloride	20-50 "
pH (glass electrode)	0-0.5
Temperature	50°C.
Current density	10 amp./sq. ft.

The palladium chloride may be prepared with a porous pot or by dissolving in aqua regia, followed by boiling to remove excess acid. Palladium anodes are used and the anode and cathode deficiencies are about 100%. Agitation is desirable and buffers such as oxalic, phosphoric, acetic, boric, citric, trichloracetic and monochloracetic acids may be used.

U. S. Pat. 2,335,821.

PLATINUM

The oldest known platinum solutions date back to a French patent issued to Pilet (1884). Based on this patent, Pfanhauser recommends the following formula:

Water	1 liter
Ammonium phosphate (secondary)	20 grams
Sodium phosphate (secondary)	100 "
Platinic chloride $H_2PtCl_6 \cdot 6H_2O$	4 "
(equivalent to about 1.5 grams of metal)	
Voltage 3-4; current density	9.3 amp./ft.^{2} (1 amp./dm.^{2})
Temperature	70-90°C.

This solution has to be boiled until the original orange color has turned into a pale yellow, indicating the formation of a complex ammino-phosphate. For bright flash deposits, a pH of 7 or slightly below is recommended. For heavier deposits, an excess of ammonia is advisable.

The rate of deposition is rather low, about 8 milligrams per amp. minute in a new bath. The efficiency drops with use to about 4 mg. per amp. minute.

This solution is replenished by the addition of platinic chloride and of course has to be boiled after each addition keeping it ammoniacal all the time until the color is a pale yellow.

Due to the rapid accumulation of chloride, the useful life of this bath is very short.

Better results are obtained with the so-called "Platinum P-salt," a platinum diammino nitrite of 61% metal content.

Keitel & Zschiegner recommend the following formula: (Baker & Co., U.S.A. Patent No. 1,779,457; U.S.A. Patent No. 1,779,436).

Ammonium nitrate	100 grams
Sodium nitrite	10 "
Platinum as diammino nitrite	10 "
Ammonia	50 cc.
Water	1 liter
Voltage 4.5; current density	55-120 amp./ft.^{2} (6-13 amp./dm.^{2})
Temperature	203°F. (95°C.)

This solution covers very quickly and the throwing power is fair. For the building up of heavier deposits, a lower voltage of 2.4 volts and 55 amp./ft.^{2} (6 amp./dm.^{2}) is recommended.

The bath is replenished with platinum P-salt dissolved in ammonia and no accumulation of salts takes place since the nitrite radical with ammonium nitrate forms water and nitrogen which is given off in gas form. These solutions have an almost unlimited life if kept free from contamination with base metals.

RHODIUM

The use of straight rhodium chloride or sulphate does not yield very satisfactory plating solutions. Rhodium for plating purposes is usually sold in form of ready prepared concentrated solutions which in most cases contain the metal in form of an acid phosphate complex.

Two types of solution are most widely used:

1. The Sulphate Type:

Sulphuric acid, conc.	35 grams (about 20 cc.)
Metallic rhodium as prepared solution	2 grams
Water	1 liter
Temperature 110-120° F., current density	10-80 amp./ft.^{2}
Voltage	2½ to 5

2. The Phosphate Type:

Phosphoric acid, 85%	10-40 cc.
Rhodium metal in prepared form	2 grams
Water	1 liter

Current and temperature conditions are approximately the same as for the sulphate type bath. This latter type of solution is preferred in all cases where white metal (tin alloys) or soft soldered merchandise is to be plated.

Insoluble anodes of platinum are used with this bath and the replenishing is done by adding the prepared rhodium solution to the bath. This procedure, of course, will gradually increase the acid concentration of the bath. This, however, has no bad effects except a slight decrease in the rate of deposition.

The rate of deposition varies from about 2 to 4 or 5 mg. per amp. min. according to the manufacturers' specifications.

In all cases, a good nickel deposit should be applied before rhodium plating. This is particularly important on white metal (tin alloys) and soft solder; otherwise a dark and streaky deposit will result.

A substantial nickel underplate increases the wearing qualities of rhodium plated articles which are subject to hard use or high temperatures.

In most cases, the rhodium deposit is only a light flash plate. In cases where better wearing qualities are desired, 1 milligram of rhodium per sq. inch seems to give good results; such deposit would have a thickness of about 0.000005".



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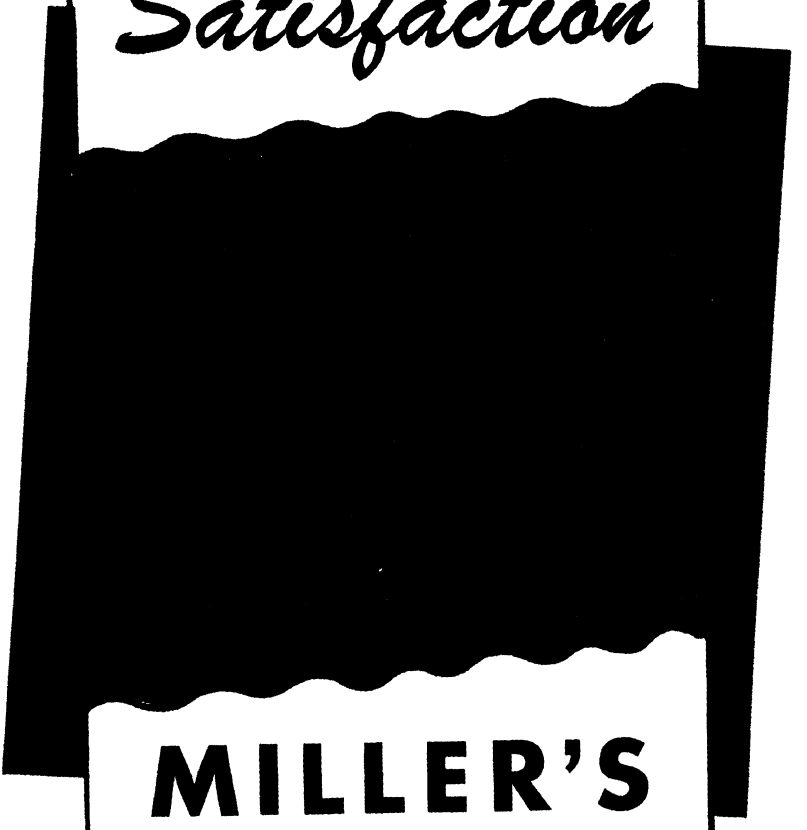
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SILVER PLATING

By N. E. Promisel

Bureau of Aeronautics, Navy Dept., Washington, D. C.

GENERAL

Silver can be plated from a variety of baths, such as the thiocyanate, thiourea-nitrate, and iodide, but except in special cases, the cyanide type is universally used. The fundamental constituents of the latter are silver, sodium or potassium cyanide in excess of that needed to keep the silver dissolved (called "free cyanide"), and either potassium or sodium carbonate. More recently, the addition of potassium nitrate has been used in some cases to replace the carbonate. A solution containing these substances, when properly operated, gives smooth, adherent, matte white deposits. To secure semi-bright deposits, carbon disulfide is generally added. Other brighteners have been recommended but they are not in general use.

It is customary to use two baths for silver plating. The first—called the "strike" solution—has a very low silver concentration and a very high free cyanide content, and is used merely to get the first covering of silver, after which the silver is built up in the "plating" solution which is as described above. The "strike" solution probably serves several functions, including the prevention of deposition of silver by "immersion" with resultant poor adhesion of the ultimate deposit, improvement of covering power and indirectly of throwing power, and cleaning. While it is possible to avoid the use of a strike solution in some cases, it is definitely not advisable, nor, on the other hand, is it efficient practice to attempt to build up appreciable thicknesses of silver from a "strike" solution rather than from the legitimate "plating" solution.

The anodes used for silver plating should be the purest obtainable, in general assaying over 999.5 fine. Very small traces of impurities, such as bismuth, manganese, lead, iron, antimony, selenium, tellurium, palladium, etc. of the order of hundredths of a per cent or even less, may cause the anodes to be covered with a blackish scum, which not only interferes with normal anode corrosion but may cause rough plate due to migration of the sediment to the cathode. Anode filter cloths and bags help in this respect. Increasing the free cyanide is also often beneficial. In any case, however, "black anodes" are an unnecessary evil. The corrosion of the anodes, as well as the tendency for blackening, depend very closely on the free cyanide content of the solution and on the anode current density. For best results, the former should be ample, as already indicated and as given in the formulas below, and the latter should be kept low by making the ratio of anode to cathode areas not less than 1:1. The carbonate content is also of importance for proper corrosion of the anodes and in fact determines to some degree the optimum cyanide content.

For contact with the solution, as for tanks, anode racks, cathode racks, etc., iron is a convenient and satisfactory material, especially if its original tight black scale has not been pickled off. It is important to keep the solutions free from chlorides to prevent attack of this material, which attack is sometimes accompanied or followed by deposition of silver on the material itself. Ceramic material and glass may also be used in some cases, but except for small tanks, these tend to be impractical because of their greater cost and marked fragility,



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which is an important item in the storage of the relatively expensive silver plating solutions. Several synthetic plastics also appear to be satisfactory, of which the phenol-formaldehyde type and certain polyvinyl compounds have been tested with good results.

FORMULAS & PLATING SOLUTIONS

Good deposits of silver may be obtained under widely varying conditions. The following formulas are recommended, not as being exclusive, but as being most satisfactory for general purposes. Non-ferrous metals require treatment only in the second strike before plating. Iron and steel require treatment in a first or steel strike until coated, after which they are treated in the second strike and plated.

Strike Solutions

	First (Steel)	*Second*
Silver cyanide	0.25 av. oz./gal.	0.5- 0.7 av. oz./gal.
Copper cyanide	1.5 " "	--- -- " "
Sodium cyanide	10.0 " "	8.0-10.0 " "
Free cyanide	8.0 " "	8.0-10.0 " "
Metallic copper	1.0 " "	--- --- " "
Metallic silver	0.2 tr. "	0.4- 0.5 tr. "

Room temperature, 6 volts, 15-25 amp./sq. ft.

Plating Solutions

I.

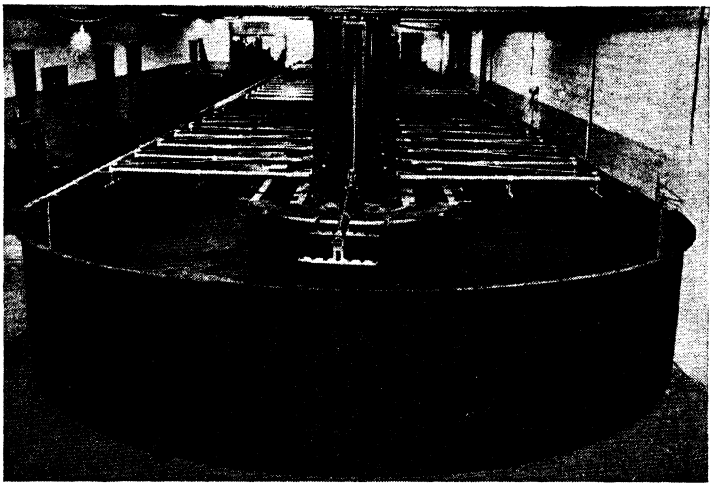
Silver cyanide	4.0 av. oz./gal.
(Metallic silver equivalent of above)	3.0 tr. "
Potassium cyanide (free)	5.5 av. "
Potassium carbonate	6.0 " "
Brightener (in a new solution)	of the order of ⅛ fluid oz./gal.

II.

Silver cyanide	4.0 av. oz./gal.
(Metallic silver equivalent of above)	3.0 tr. "
Sodium cyanide (free)	4.0 av. "
Sodium carbonate	6.0 " "
Brightener (in a new solution)	of the order of ⅛ fluid oz./gal.

III.

Silver cyanide	3.2 av. oz./gal.
(Metallic silver equivalent of above)	2.3 tr. "
Sodium cyanide (free)	2.5 av. "
Sodium carbonate (maximum)	As low as possible
Potassium nitrate	15 av. oz./gal.
Brightener (in a new solution)	of the order of ⅛ fluid oz./gal.
Brightener (in a new solution)	of the order of ⅛ fluid oz./gal.



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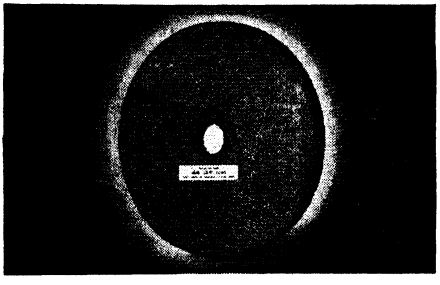
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For all formulas:

Temperature 72-78°F., preferably the latter
Current density 5-15 amp./ft.^{2}
Voltage—From less than 1 volt to a few volts, depending on operating conditions, solution content and geometry of the set-up. For most small set-ups, the voltage is about 1 volt.

The brightener may be made by shaking a few ounces of carbon disulfide with a quart of "strike" solution and allowing it to stand for a few days, with occasional stirring. Only the clear supernatant liquid is used as a brightener. The amounts of brightener to be added to an old solution cannot be specified. Only trial-and-error methods, guided by experience, can determine these quantities, the correctness of which can be confirmed only by the appearance of the deposits themselves.

Ammonium thiosulfate has recently become readily available and found wide acceptance as a brightener. For a new plating solution 1.25-2.5 fluid ounces of 60% ammonium thiosulfate solution are used to each 100 gals.

The potassium solution above (No. I) is the most popular and is to be preferred over the sodium cyanide solution for commercial plating, even though somewhat more expensive than the latter. The recommended values are very commonly used, although for light plate, especially in great volume, the lower ranges are employed, thus reducing the cost of the solution and the amount of silver dragged out. Formula No. III (Wood's bath) is now being used in some plants. It is less expensive than the others and gives excellent results. The full story of its advantages and limitations has yet to unfold as experience with it grows. Silver nitrate may be used to make it, instead of silver cyanide, changing, proportionally, the amounts of the silver salt and total cyanide used.

Effects of Various Factors on the Solution Properties and Operation

The effects of the various factors can be briefly summarized as follows:

To increase throwing power: Decrease silver or brightener content, increase cyanide or carbonate contents, increase current density, lower temperature.

To decrease resistivity of bath (e.g. lower bath voltage or sometimes to increase throwing power): Increase temperature, cyanide content, silver content, or carbonate content.

To increase permissible current density: Increase silver and free cyanide content, increase temperature, increase brightener, increase agitation, use potassium solutions, increase uniformity of plate by increasing throwing power and by improving method of racking. Increasing the temperature from 70° to 90°F. may allow a 50% increase in current density.

To reduce anode polarization: Increase temperature, decrease anode current density, decrease silver and carbonate content, and increase free cyanide.

Treatment of Work Before and After Plating

The work before silver plating must naturally be properly cleaned, but this may be done in any of the usual satisfactory routines. In plating copper, brass, Britannia metal, soft solders, or similar alloys, a mercury dip is often employed, containing for example about ⅓ oz./gal. mercuric nitrate and hydrochloric acid

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to make the Baume reading about 7. This solution then replaces the usual acid dip. Mercury dips should be used with care since they are prone to stain the metal. They are, in fact, advisedly omitted when it is possible to do so and this omission is usually possible. High brasses are particularly prone to "season cracking" and this difficulty is accelerated by mercury. It is desirable to conclude the preplating dips with a dip in sodium cyanide (10 oz./gal.) from which the work may be directly transferred to the strike bath without rinsing. A rinse between strike and plating solutions is optional, each method possessing certain merit. Rinsing diminishes the danger of dragging into the plating solution any chloride or iron which might be present in the strike solution. On the other hand, if the rinse is used, valuable cyanide and silver are lost to the plating solution the cyanide being, furthermore, irretrievable.

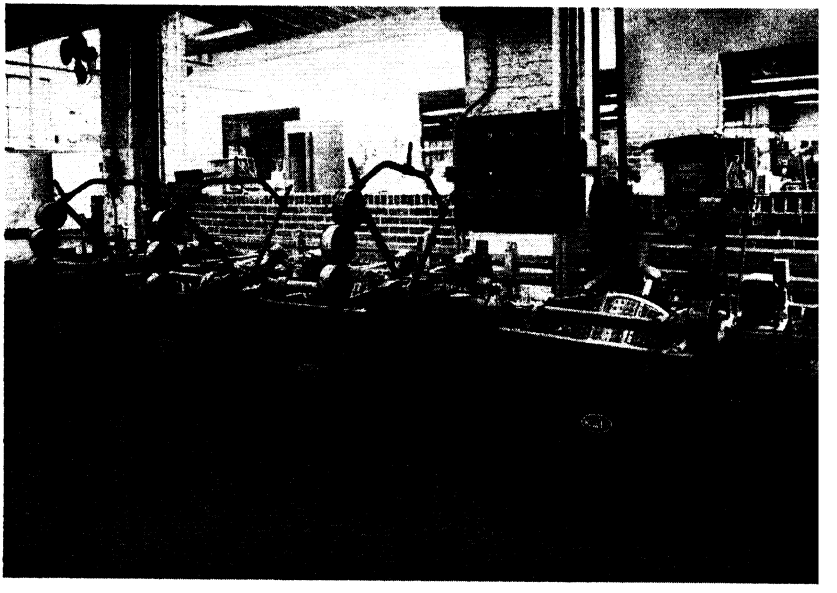
After plating, the work should be rinsed in two cold water rinses and one hot water rinse. The former two are stagnant but are replaced frequently depending on the volume of work going through them. They are not thrown away but are collected in a large tank and the silver reclaimed for shipment to the refiners by adding excess hydrochloric acid and allowing the precipitate to settle before decanting the waste liquor. (Caution: Beware of the fumes, which are very toxic.) The hot water should be a running rinse and should be kept very clean. The recovery of silver from this rinse should not be necessary if the work has been properly treated in the preceding two.

Maintenance of Solution

If proper attention is paid to minimizing drag-out and if sufficient silver anodes of required purity are used, the silver content in the bath is ordinarily maintained automatically to a very satisfactory degree, since the anode and cathode efficiencies are both essentially 100%. If silver is needed in the bath, it is best added as silver cyanide or potassium (or sodium) silver cyanide. In the case of Wood's bath, silver nitrate may be used. The free cyanide in the bath will gradually decrease on operation and standing, and must be replenished The brightener should be added in small amounts at least once or twice a day for steady production. This component disappears from the bath even when the latter is not in operation. Decomposition products, such as formates, ammonia and its compounds, and a variety of organics, also exist in the bath, but little is definitely known about their specific effects and apparently the bath can usually function very well with them. As far as metallic impurities are concerned, perhaps the greatest danger is from iron, because of the amounts which can be accumulated if the plating room is carelessly operated. Of greater effect are some foreign anions. In general, however, none of these unintentionally present substances become of importance in a properly maintained solution.

The carbonate content of the solution will gradually increase to a value determined by drag-out losses. In a solution containing only potassium salts, a carbonate content of 15 oz./gal. will still give satisfactory results. In a "sodium" solution, this figure should be reduced by a few oz./gal. at least. For removal of carbonate, several methods have been suggested. "Freezing out," as in cold weather, is one suggestion but this is rarely effective in the case of potassium solutions (due to the appreciably solubility of potassium carbonate even at low temperatures) and in the case of sodium solutions, the method is more effective but it is inconvenient. Precipitation by barium cyanide is expensive. Gypsum has been recommended as a precipitating agent but not wholly satis-

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factory results have been obtained so far and this material should be used cautiously. The above difficulties are not, however, of really great concern, because if potassium solutions are used, the carbonate rarely reaches a concentration which is seriously troublesome. When nitrates are used in the bath, calcium nitrate is used to remove the carbonate.

TIN PLATING FROM ACID BATHS

By F. C. Mathers
Indiana University, Bloomington, Indiana

SULFATE SOLUTION

The tin deposits from acid solutions are whiter, but are often less adherent to the basis metal than are the deposits from alkaline baths. Of course, the throwing power of the alkaline baths is much better than that of the acid baths. However, the throwing power of the acid tin baths is better than that of most non-alkaline plating baths now in general use. On the other hand, tin can be deposited two to three times faster from acid baths than from alkaline baths. This is due to the higher cathode efficiency, 100% compared to 75%, and to a valence for tin of 2 compared to 4 for the alkaline baths. Also, the acid baths will operate nicely at room temperature, whereas the alkaline baths must be heated. The acid baths have very high electrical conductivity and the anodes dissolve with such high current efficiency that maintenance of the baths is not a problem, except for oxidation of the stannous to stannic by the air whereby a sludge is usually formed.

NOTE: Electroplated tin on iron has about the same porosity as a hot dip coat twice as thick.

No "bright" tin bath is known although semi-bright deposits may be obtained at the higher current densities. It is the common practice, if a bright finish is desired, to heat the plated articles sufficiently to melt the tin. The basic patent covering this has expired.

Most of the soluble tin (stannous) salts can be used in plating baths except chloride, bromide, and iodide, and these ions are injurious in any of the other baths. However, it is reported that "halogenated" tin baths are being tried but no information regarding the composition or addition agents has been made public.

Usually the stannous sulfate bath is used, because stannous sulfate is the cheapest tin salt that will give satisfactory deposits. Several companies now manufacture an excellent quality of the stannous sulfate and the prices are moderate.

Stannous fluoborate and fluosilicate do not give enough better results to justify their greater cost, although their greater solubilities allow more concentrated solutions to be used, and the deposits are distinctly less coarsely crystalline. The fluoride bath is less desirable than the sulfate because the deposits are much coarser. However, fluoride baths develop much less sludge or precipitate than any of the others.

The writer does not recommend phenol- or cresol-sulfonate in the baths although many formulas use these materials.

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NOTE: It is claimed that the cresol-sulfonic acid prevents the formation of sludge during operation.

A recent publication in a foreign journal[4] clearly states that cresols and phenols—called surface active agents—should be used in the unsulfonated form, and more of them—20-30 g./l. of cresol—than recommended in any of the following formulas. However, another recent publication recommends the use of a large quantity of cresol-sulfonic acid like that in formula No. 3.

There are few published descriptions[1] of the most important researches on tin plating, but certain patents [Mathers, U. S. Nos. 1,397,222 and 1,540,354; Simpkins, U. S. No. 1,452,273 (1923); Pine, U. S. No. 1,987,749 (1935); and Brit. 493,701 (1938)] give some accurate information. Publication No. 92 by International Tin Research and Development Council, obtainable by addressing Battelle Memorial Institute, Columbus, Ohio, contains an extended discussion of electrotinning. The best recent general discussion of acid tin plating is the paper by Pine.[2]

A recommended formula is:

Acid Sulfate Tin Solution

	oz./gal.	g./l.
Tin Sulfate, $SnSO_4$	13	100
Sulfuric acid, H_2SO_4, Conc.	4	30
Tartaric acid	4	30
Glue	0.4-0.8	3-6
Cresol or	0.8	6
β-naphthol	0.13	1

Pine suggests the following ranges in composition:

	oz./gal.	g./l.
Tin sulfate	4-12	30-90
Sulfuric acid	7-20	50-150
Gelatin or glue	0.06-1.3	0.5-10
Cresol	0.06-1.3	0.5-10
Sulfonated cresol	1.3 -13	10-100
β-naphthol	0.06-.13	0.5-1
Resorcinol	3	20
Sodium sulfate	7-50	50-350
Aloin	0.13-0.4	1-3

He says not more than three of the addition agents need be used at one time, but gelatine or glue should always be one of them. As a specific example of a bath, he gives:

	oz./gal.	g./l.
Stannous sulfate	7	54
Sulfuric acid	13	100
Cresol sulfonic acid	13	100
β-naphthol	0.13	1
Gelatine or glue	0.3	2

The criticism the writer offers to this formula is that the tin concentration is too low for best deposits, but, if drag out is very large, such a formula would be more economical than the first one favored by the writer.

Without addition agents, these acid baths could not be used at all, as the

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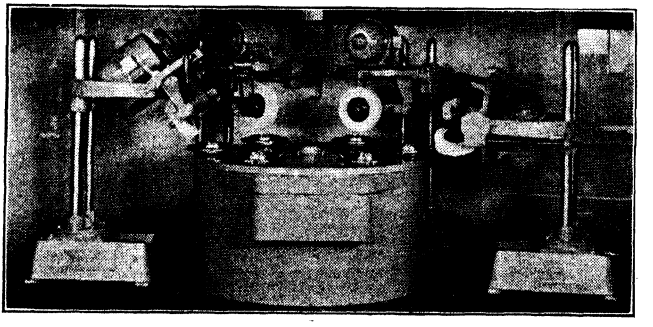
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deposits would be masses of loose crystals. No single addition agent will suffice, and the best combination is glue and cresol, or cresylic acid, or other pairs of organic compounds belonging to these two families. The composition of the baths can be varied through wide limits without much change in results. However, the more concentrated the solution, the higher the current density or voltage that can be used, but, in all cases, the lower the current density or voltage, the better the deposits. The rather low solubility of stannous sulfate, 180 g./l., (purified stannous sulfate has a higher solubility—over 300 g./l., according to Richter) strictly limits the maximum concentration that can be used, and the sulfuric acid and any sodium sulfate in the solution further reduce this solubility by common ion effect.

A lower concentration of tin sulfate, sulfuric acid, and tartaric acid, in the above proportions, may be used at the lower currents. However, it is best to use the full quantities of the cresol or β-naphthol and glue, and this writer favors using just as large quantities of these addition agents as is possible without causing too much brittleness of the deposit.

Animal glue, not vegetable glue, must be used. The glue should be soaked in water for a few hours and then be brought into complete solution by heating before being added to the bath.

The most serious objection to β-naphthol is its very limited solubility in water—0.74 g./l.—and therefore, not enough is present in solution to do the work. The bath can be saturated with β-naphthol by adding to the bath a solution of β-naphthol in alcohol, in which it is very soluble.

The baths should be run at room temperature and a maximum current of 46 amp./ft.^{2} (5 amp./dm.^{2}) is recommended although it is possible to use 400 amp./ft.^{2} (43 amp./dm.^{2}) with some baths, where there is movement or agitation of the cathode and where only thin deposits are being made. The voltage varies from 0.6 to perhaps 3 or 4 depending upon the current and the size and the purity of the anodes. It is best to use the lower currents, except for the very thinnest deposits, and lower currents must be used if thick deposits are desired. The exceptionally pure tin, produced by some of the detinning companies, should be used for anodes. Such secondary tin is very much purer than the "straits" tin, and less sludge and anode cake or slime will be produced. The sludge from the anodes of crude South American tin would cause difficulties. Any hard crust that may form on the anodes must be brushed off. This will not be necessary unless impure anodes are used. If only impure anodes are available, the use of a fluoborate in place of part of the sulfate would very much lessen anode difficulties. Lead or rubber lined tanks can be used.

Although it is better to prepare the plating solution from purchased stannous sulfate, the baths can be prepared from tin anodes by the porous pot method using a solution of sulfuric acid containing 10 oz./gal. (75 g./l.). About 26 ampere hours are needed for each liter (108 amp.-hr./gal.) of solution. This seems to be the usual method in England, and the refining baths during World War I were prepared in this way. This method must be used for making tin fluoborate, fluosilicate, or cresol-sulfonate baths, as these salts are not available on the market.

A poor quality of cresol (crude grades are called cresylic acid) contains oils which are insoluble in the bath. The part of the cresol which does not dissolve, floats on the surface of the bath, and smears the work as it is being put in for plating. Some method of keeping the bath saturated with the cresol without this surface layer of oil should be devised.

Sludge forms in the baths due to residues from the addition agents and to

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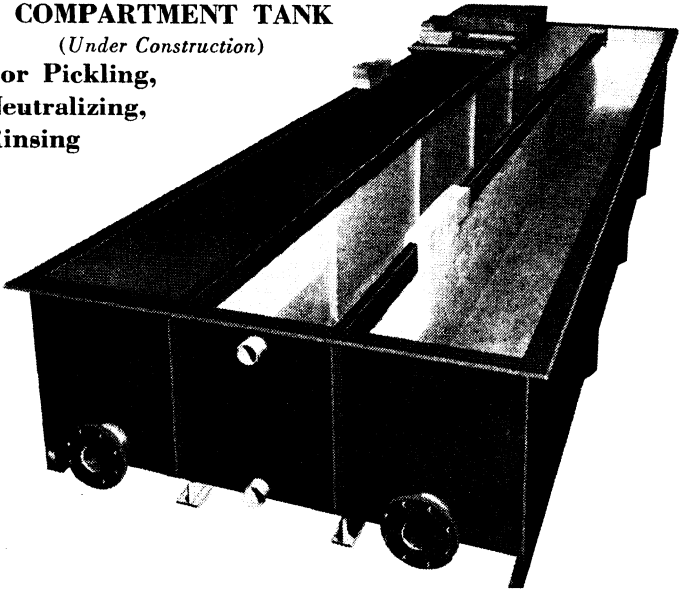
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insoluble basic stannic salts which are formed by air oxidation. Pine says that filtration is not very necessary unless exceptionally impervious deposits are desired, and that satisfactory deposits can be obtained, for most purposes, even when considerable suspended matter is present. The tartaric acid in the formula lessens the formation of sludge. Hydrofluoric acid (4 oz./gal.) will also restrain sludge formation, but glass and earthenware vessels and apparatus would be corroded by it. Some formulas use cresol-sulfonic acid but this cresol is not active as an addition agent, and, therefore, the required quantities of unsulfonated cresol or β-naphthol must always be added regardless of whether any cresol-sulfonic acid is used.

Several methods of making stannous sulfate for plating baths have been published.[3] Shot or granulated tin cannot be dissolved in hot sulfuric acid for use in making these baths, but spongy tin or tin foil will dissolve. Any form of metallic tin will dissolve in dilute sulfuric acid or in the bath itself if the tin is alternately exposed to the air and immersed in the solution. A better way is to rotate the shot or granulated tin in a tumbling barrel with only enough sulfuric acid that the tin is partly in the air all the time. This is a very economical way of making a plating solution or increasing the metal concentration of a depleted bath.

More cresol or naphthol, and glue must be added as needed, and the indications of such a need are (1) a decrease in voltage of 0.2 under that for a new solution, (2) a visible increase in grain size, or extending crystals or roughness at the corners or on the edges, and (3) a decrease in covering power.

Cresol-sulfonic acid, needed for any of the formulas, can be made without any difficulty by heating one part of cresol with 2 parts of conc. sulfuric acid for 2 hours at 230°F. (110°C.).

1. Stack. Trans. Electrochem. Soc., 45, 441 (1923).
2. Pine, Trans. Electrochem. Soc., 80, 681 (1942).
3. Mathers and Rothrock, J. Ind. and Eng. Chem., 23, 831 (1931).
4. Chem. Abstracts, 35, 2421 (1941).

TIN PLATING FROM ALKALINE BATHS

By Floyd F. Oplinger

E. I. dul'ont de Nemours & Co., Inc., Wilmington, Delaware

SOLUTION COMPOSITION

	Still Tank	Barrel
Sodium stannate	16	20 oz./gal.
Sodium hydroxide	1	2 "
Sodium acetate	2	3 "
100 vol. Hydrogen peroxide or Sodium perborate	1/16	1/16 "

In order to prepare the solution, fill the tank with water to two-thirds its capacity and heat to 140-158°F. Add the required amount of sodium stannate, slowly, at the same time stirring thoroughly until all is dissolved. Then add the weighed amount of caustic soda required. When completely dissolved, add the required amount of sodium acetate. Then add the balance of water required to bring the bath up to the operating level.

Before adding the peroxide or sodium perborate, it is advisable for control

purposes to determine the tin and "Free Caustic Soda" content. Considerable care should be exercised to adjust the caustic soda content by analysis to conform to the limits allowed (see following page). After this has been done, the sodium perborate or hydrogen peroxide should be added and the solution is then ready for use.

Caution

Caustic soda is formed by hydrolysis of the stannate, and some is present in the stannate. Therefore, to avoid an excessive concentration of caustic soda, the bath should first be prepared according to the directions and if analytical facilities are available, the composition should then be adjusted to the concentrations given. One of the reasons for close control is to maintain anode efficiency so that it will not be necessary to replenish with large amounts of stannate from time to time.

FUNCTIONS OF THE BATH INGREDIENTS

Sodium Stannate

The sodium stannate is a reservoir for the tin which is deposited at the cathode. Maximum efficiency is obtained only when the sodium stannate content is greater than 6.0-8.0 oz./gal.

Sodium Acetate

The function of the sodium acetate is similar to that of boric acid in a nickel solution, that is, it acts as a buffer. It also aids in increasing anode solubility.

Hydrogen Peroxide or Sodium Perborate

These oxidizing agents serve as a means of keeping the tin in solution in the quadrivalent form. Stannous ions, when present in appreciable amounts, are harmful, causing the deposition of spongy tin.

A summary of the operating conditions most generally useful is given in the following table.

Summary of Plating Conditions

	Still Solution	Barrel Solution
Cathode current density	6-60 amp./ft.^{2}	
Anode current density	5-25 "	as low as possible*
Voltage	4.0-6.0 volts	not less than 6 volts*
Temperature	140-176°F. (60-80°C.)	160-180°F. (71-82°C.)

*This means the use of as large an anode surface as possible, because it is desirable to use the highest possible amperage.

Caustic Soda

The caustic soda acts as a conducting agent and is absolutely necessary to obtain balanced anode corrosion. Without caustic soda, the tin anodes become coated with a film which interferes with proper anode reduction. In consequence, the tin plates entirely from the solution which thus becomes depleted. On the other hand, an excess of caustic soda causes the deposition of rough and spongy deposits and poor throwing water.

The caustic soda content, determined with Sulfo-Orange indicator, should be between 1.0 and 2.5 oz./gal., preferably 1.6 in still tanks, and between 2.5 and 5.0 oz./gal., preferably 3.0 in barrel solutions.

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CONTROL AND MAINTENANCE

Anode Current Density—Still Plating

Pure tin anodes should be used. One of the most important factors for the successful operation of the bath is proper control of anode current densities. In order to reach the proper anode current density for any given bath or set of operating conditions, the anode current density must be sufficiently high so that the anodes become oxidized or polarized. This condition is easily recognized because it occurs when the anodes become covered with a greenish yellow film believed to be a mixture of stannous and stannic oxides. This yellow film has a very characteristic color and is easily recognized when once seen. Satisfactory results can be obtained only when the anodes are covered with this film. When the anode current density is too low, the anodes remain clean and may be covered with a light, loosely adherent film of dark or black particles and the voltage normally used drops. At this point, the natural color of the tin crystals is clearly visible. When the anode current density is too high, oxygen is given off, little or no tin is dissolved, and the anodes become covered wholly or in part with a black oxide film which becomes highly insoluble and is removed only with strong mineral acids.

Deposits may remain satisfactory for a time but the metal content becomes depleted rapidly, following which dark rough deposits are produced. Usually an anode to cathode ratio of 1 to 1 is satisfactory. If anodes become scaled or covered with sludge, caustic soda should be added.

The bath can be controlled for long periods of time by observing the above rules.

For barrel plating, the anode surface area should usually be as large as it is possible to obtain in equipment being used. In barrel plating, in order to keep the anodes clean and properly polarized with the greenish yellow film, it is usually advisable also to use a higher caustic content than for still plating.

The anode current density and the caustic soda content should always be closely controlled in both still and barrel plating.

With proper precautions, anode efficiency can be kept equal to or slightly higher than cathode efficiency. This means that the most of the metal can be obtained directly from the anode which is a more economical way of doing than keeping the metal content up by means of addition of sodium stannate.

Cathode Current Density

Excellent results may be produced over a wide range. For practical purposes 15-25 amp./ft.^{2} give the best results.

Sometimes solutions are operated in closed areas where ventilation is poor. Under such conditions the evolution of hydrogen from the bath may become very objectionable to persons working in the vicinity of the solution. This condition may be prevented by adding small quantities of sodium oleate or other soluble soap to the solution. One pint of soluble soap is enough for several hundred gallons of solution. This treatment causes the formation of a foam on the bath which prevents the dispersion of the fumes through the surrounding atmosphere.

Rough Deposits

Generally speaking, rough deposits are caused by having an excess of stannous ions in the bath. An excess of such ions usually affects the color of the bath.

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Normally, it should be a light straw or gray color. If the solution looks black or dark, trouble can be expected. Stannous ions decompose spontaneously with the formation of sponge tin which travels to the cathode causing roughness. The condition can generally be immediately corrected through the addition of a small amount of hydrogen peroxide or sodium perborate.

This, however, may be only a temporary remedy. If the objectionable condition re-occurs soon after the addition of peroxide, then the anode current density is probably too low or the caustic soda concentration is too high. When the caustic soda concentration is too high, it may best be reduced through the addition of small amounts of acetic acid. Acetic acid should be diluted considerably before adding to the bath and when added, should be added slowly with constant stirring. Mineral acids should not be used when reducing the caustic soda content because they cause the precipitation of basic tin salts.

Commercial size solutions should not be operated for appreciable periods of time with less than full tank loads unless some of the anodes are removed. The reason for this is that if only a small cathode surface is being plated, the anode current density will be very low when the tank is full of anodes. This produces stannous ions and spongy or rough deposits may follow.

The most important factors to watch are the caustic soda content, and the anode current density. Tin will dissolve in the stannic condition only when these are properly balanced.

Temperature of Solution

For still solutions, temperatures ranging from 140 to 176°F., that is 60-80°C., give consistently good results. At lower temperatures, the deposits may become dull and the solution loses efficiency. Higher temperatures should not be used because of the danger of precipitating insoluble tin salts.

In barrel plating it is advisable to operate in the temperature range 160-180°F. This slightly higher temperature improves the conductivity of the solution.

Replenishing the Solution

Never add undissolved material to the solution. Any material which needs to be added should be first dissolved in water and the aqueous solution added slowly with constant stirring.

Time of Plating

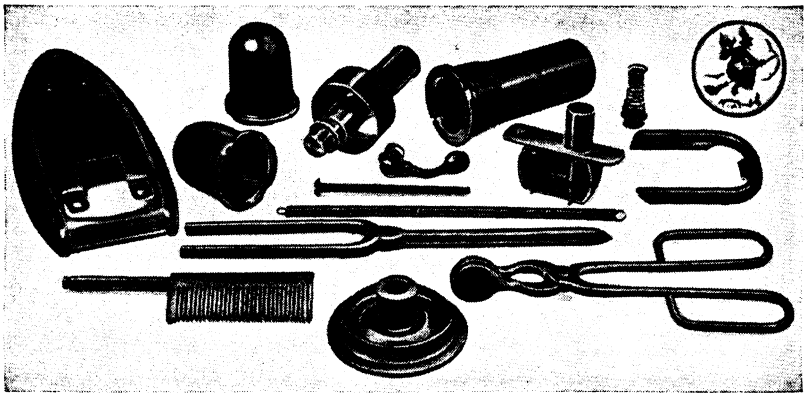
In general, it is safe to assume that one ampere deposits one gram of tin per hour so that at 25 amp./ft.^{2}, 0.0147 oz./ft.^{2} of tin is deposited per minute. For a coating of 0.294 oz./ft.^{2} or 0.00048", twenty minutes plating time is required.

For producing non-porous deposits on steel, a thickness of not less than 0.00075" is generally required. This may be more or less depending on the condition of the base metal.

Equipment Required

For still solutions a clean steel tank equipped with steel or iron-heating coils is preferred.

For barrel solutions the tank also should be of steel. Wood or most synthetic plastic cylinders are not suitable for horizontal type units. The plating barrel cylinder should be constructed of material recommended by manufacturers of barrel plating equipment used for this purpose.



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Water

Wherever possible, distilled or softened water should be used for preparing solutions. The same holds true of water required to keep solutions up to the proper level. Water hardness is one of the main sources of sludge formations. Baths can be operated with soft water so as to remain virtually free from sludge. A simple means of softening the water, if better means are not available, is to add small amounts of caustic soda (about ½ lb./100 gal.) to water in a separate container. When the precipitate has settled, decant the clear water to the plating tank. The use of soft water for plating solutions becomes more and more important as time goes on.

ACID ZINC PLATING

By R. O. Hull

R. O. Hull & Co., Cleveland, Ohio

Acid zinc plating is widely used for plating of wire, strip steel, wire cloth and pipe, which do not require a bright deposit, but a smooth, white, rust-resistant coating. Acid zinc solutions are not applicable to plating of irregular articles, because of their inferior throwing power. Thompson's solutions which follow cover the usual range of application of this type of solution.

SOLUTION COMPOSITION

	Solution No. 1 oz./gal.	Solution No. 2 oz./gal.
Zinc sulfate (crystals)	32	48
Ammonium chloride	2	4
Aluminum sulfate (crystals)	4	—
Sodium acetate	—	2
Licorice	.13	—
Glucose or corn syrup	—	16

Operating Conditions

Metallic zinc	7.2	11.0
Temperature	75-120°F.	
pH, still solution	3.5-4.6 (electrometric)	
pH, barrel solution	5.0-5.3	
Corresponding current density	5-50 amp./ft.^{2} still.	
	50-100 " agitated.	

or

Acidity	0.1-4.0 N (by titration)
Corresponding current density	100-500 amp./ft.^{2} agitated

Tanks—rubber or lead lined.

Anodes—good grade intermediate zinc-mercury, zinc-aluminum-mercury or electrolytic.

Solution Maintenance

1. Maintain all constituents within ± 10% of above values.
2. Establish either proper pH or normality of acid for desired current density (the higher the current density, the lower the pH or higher the acidity) and maintain at this value by additions of sulfuric acid (usually) to lower

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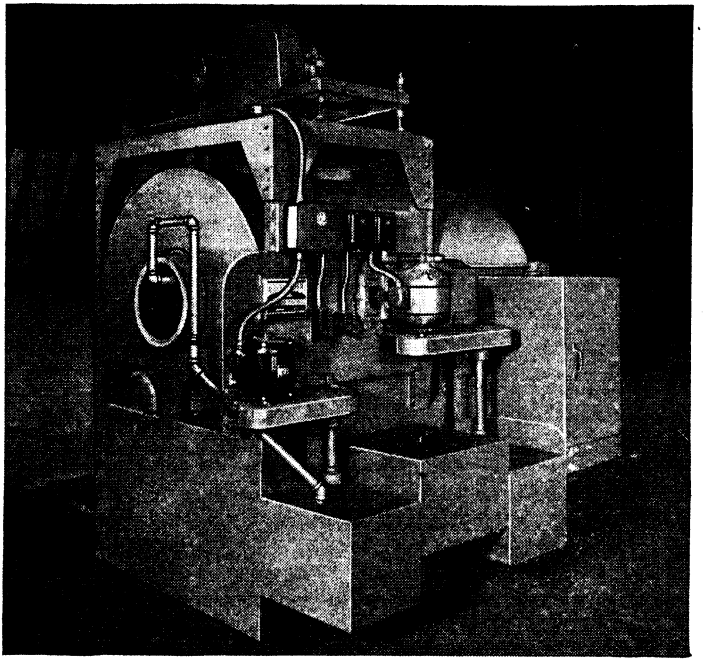
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the pH or increase acidity, or ammonia (very seldom) to elevate the pH or decrease acidity.

3. Special anodes are available as noted above for acid zinc which corrode more uniformly with less sludge than straight zinc.
4. Add brighteners as necessary.
5. For deposits over 0.0002" thick, bag anodes and filter the solution.

CYANIDE ZINC PLATING

By R. O. Hull

R. O. Hull & Co., Cleveland, Ohio

SOLUTION COMPOSITION

	Cyanide Zinc (Blum & Hogaboom) oz./gal.	Bright Zinc Still or Barrel oz./gal.	Mercury Zinc (1) Still oz./gal.	Mercury Zinc (1) Barrel oz./gal.
Zinc cyanide	8	8	5	8
Sodium cyanide	3	5.6	3	4
Caustic soda	7	10.5	4	6
Mercuric oxide (1)	—	—	.03	.03
Operating Conditions				
Metallic zinc	4.5	4.5	2.8	4.5
Total sodium cyanide	9.7	12.3	7.2	10.7
Ratio of **total** NaCN/Zn	2.16	2.74	2.6	2.4
Temp. degrees F.	105-120	75-85	100-108	100-108
Current density amp./ft.^{2}	10-20	5-50	15-25	10
Cathode efficiency, per cent.	95	90	85-95	85-95
Anodes	Zinc	Electrolytic Zinc or (3) zinc-magnesium.	Zinc-mercury (1) or zinc-aluminum-mercury (2).	

Bright zinc usually requires patented purifiers, addition agents and bright dips. Tanks—either rubber lined or plain steel.

SOLUTION MAINTENANCE

A. Cyanide Bath Without Addition Agents

1. Maintain the solution composition within ± 10% and the ratio of NaCN/Zn from 2.0 to 2.5. The bath has good throwing power and covering power, producing zinc deposits of good quality and high ductility.

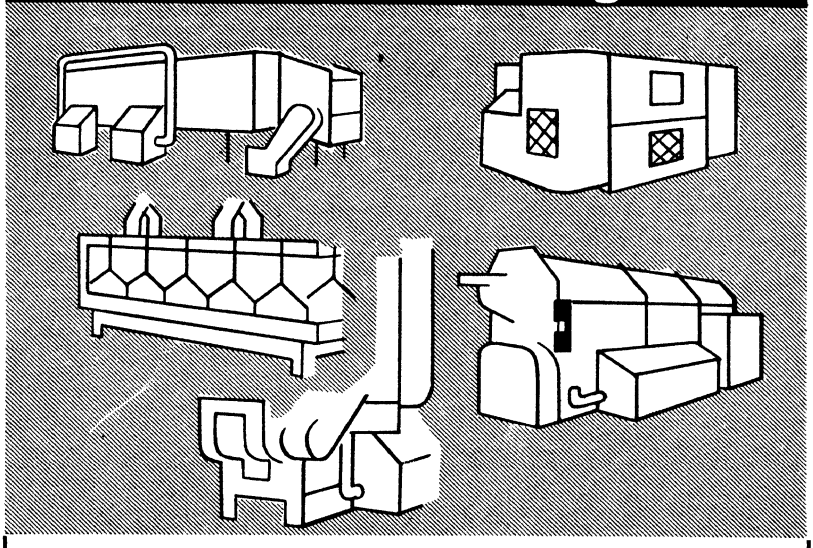
2. For fast plating, use a bath 50% stronger than given in the above formulation at a temperature of 105-155°F. and a current density within the range 55 to 110 amp./ft.^{2}

B. Bright Cyanide Zinc Baths

1. Use high purity plating materials and anodes. Lead in solution may be precipitated by treatment with a concentrated solution of sodium sulfide (2.5 g./l. or ⅓ oz./gal.). This should be stirred into the bath whenever the deposit becomes dark. The bath should be treated with the same amount of zinc dust followed by stirring and filtering if lead is excessive or copper is present.

2. Maintain the ratio of total NaCN/Zn as follows:

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Ratio NaCN/ZN	Degree F. of solution
2.6	82- 87
2.8	88- 92
3.0	93-100

The bath components should be kept at ± 5% of the concentration values given for bright deposits and high cathode efficiency. A low ratio of NaCN/Zn raises the bright current density range, while increasing the ratio lowers the bright current density range and lowers the cathode efficiency.

3. An oxidizing bright dip as 0.25-0.5% nitric acid may be used after plating to improve resistance to tarnish and finger staining.

4. Sodium carbonate rarely accumulates excessively but should be kept below 6 oz./gal.

5. Caustic soda must be maintained in proper concentration for good deposits and high cathode efficiency.

6. Special anodes containing magnesium or aluminum are available to insure proper rate of solution of zinc with minimum sludge.

C. Zinc-Mercury

1. Maintain at least 4 volts across plating tank bus bars; if voltage is less than 4, remove anodes until desired voltage is secured.

2. In plating, mercury deposits with zinc, but during week-end or other longer shutdown, the mercury in the bath deposits on anodes and must be replaced with mercury salts.

3. If bath builds up in zinc, replace one-third of zinc anodes with steel—preferably case hardened.

4. Avoid excessive mercury in the bath or anodes as this may result in mercury spots on the work which may develop after plating.

1. U. S. Patents 1,435,875, 1,451,543, 1,497,265.
2. U. S. Patents 1,887,841, 1,888,202, 2,064,307.
3. U. S. Patent 2,243,696.




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SURFACE TREATMENTS

COLORING OF METALS

By George B. Hogaboom

Consultant, New Britain, Conn.

Coloring of metals is an art. The art bronzer should, therefore, have a fair knowledge of chemical and metallurgical principles, especially of alloys. The formulas listed in this section have been taken from the literature, with a few exceptions. All have been produced on a commercial scale. The results obtained by any individual will depend more upon the skill of the operator, his appreciation of color and the nature of the object that is to be colored, than upon the formula for coloring.

Colors of almost unlimited variety can be given to metal by several methods—chemical, electrochemical, heat treatment and the application of lacquer and enamels.

CHEMICAL

The general term of "oxidizing" is applied to chemical coloring of metals. The oldest method and the one that will produce the largest number of shades ranging from black to a light brown as the final finish is the use of an alkaline sulfide. These sulfide salts were, at one time, known as "liver of sulphur" but now are divided into sodium, potassium, barium, ammonium sulfide or polysulfides, the latter, in the liquid state, being most generally used today.

The liquid polysulfide is diluted with water to the strength required for the color to be produced. The stronger solutions develop the color more rapidly but with weaker solutions, while the time of obtaining the color is longer, the results are better as the film produced is more dense.

As the color is produced progressively from an iridescent to a brown and finally to a black, it is necessary to "catch" the color desired. No definite instruction can be given as to the strength of the sulfide solution, the optimum temperature or the length of the time of immersion. That must be learned through experience.

The color obtained on copper and its alloys is dependent upon the composition and physical structure of the basis metal. A zinc bronze—low brass—can be given a distinctive brown color that is almost impossible to produce on copper. The colors produced on cast metal seldom match those produced on wrought metal. Often castings are made from a large percentage of scrap metal and, to obtain a color to approximate that produced on wrought metal, it may require that the casting be electroplated in an alloy solution. That is true for coloring many metals in any chemical solution.

In the coloring of high copper alloys to obtain a light brown shade, a weak solution of sulfuric acid—2%—called "sour water" is used to "set" the color. The article to be colored, after thorough cleaning in an alkali solution and rinsing well with water, is immersed in a sulfide solution for a few seconds, then rinsed in water and followed by an immersion in the "sour water" and again rinsed in clean water. The rinse used after the sulfide dip should not be used. This sequence of

operations is continued until the desired shade of brown is obtained. All sulfide produced films require either wet or dry scratch brushing.

"Relieving" to obtain a contrast of color may be done dry with a buff or with a scratch-brush, either wire or tampico, and pumice and water, or a suitable buffing compound.

All sulfide colors are susceptible to the action of the atmosphere, especially if warm and moist. They should be given a coating of a good lacquer—a slow drying lacquer often gives the best results when either air or heat dried.

Small articles may be colored in bulk, then lacquered and "relieved" in a clean barrel with a small amount of a mild abrasive and sawdust. After this operation the articles are re-lacquered.

It has been found that the use of a potassium sulfide solution gives the best results in bulk work that is "relieved" in a barrel. The colored coating is apparently softer or tougher and a good "relief" is obtained. Sodium sulfide films appear to be brittle with the result that a "patchy relief" is obtained.

In "Metal Coloring and Bronzing" by Hiorns will be found 135 formulas for coloring copper and copper alloys.

Some of the methods for chemical coloring of metals follow.

COPPER AND BRASS

Black on Brass

Copper carbonate	½ oz.
Aqua ammonia	4 "
Soda ash	¼ "
Water	1 gal.
Temperature	190°-200° F.

The copper carbonate should be as free from sulfate as possible. Freshly precipitated well washed copper carbonate is best to use. The black color is more permanent on an alloy of 80% copper and 20% zinc than on common high brass (65% copper, 35% zinc).

Blue on Brass

Lead acetate	2-4 oz.
Sodium thiosulfate	8 "
Acetic acid	4 "
Water	1 gal.
Temperature	180° F.

This will produce a blue color on a nickel deposit and also on polished high carbon steel. The color will change if not lacquered.

Antique Green on Brass

Nickel ammonium sulfate	8 oz.
Sodium thiosulfate	8 "
Water	1 gal.
Temperature	160° F.

Hardware Green on Brass

Ferric nitrate	1 oz.
Sodium thiosulfate	6 "
Water	1 gal.
Temperature	160° F.

Brown on Brass or Copper

Potassium chlorate	5½ oz.
Nickel sulfate	2¾ "
Copper sulfate	24 "
Water	1 gal.
Temperature	195°-212° F.

Bright Black on Brass

Hydrochloric acid	1 gal.
White arsenic	32 oz.
Antimony chloride	20 "

Do not add any water. Use hot.

Crystallized Brass or Bronze

Copper sulfate	8 oz.
Ammonium chloride	4 "
Water	1 gal.

Use either cold or warm. After crystallized effect has been obtained, a dip in a sulfide solution will bring out the crystal effect more distinct'y.

Light Brown on Copper or Brass

Barium sulfide	½ oz.
Ammonium carbonate	¼ "
Water	1 gal.

The color is made more clear by wet scratch brushing and repeating the dipping.

Barbedienne Bronze

Potassium sulfide	½ oz.
Red ammonium sulfide	¼ "
Yellow antimony sulfide	¼ "
Aqua ammonia	½ " (fluid)
Water	1 gal.

Wet scratch brush and repeat dipping operations.

Verde Antique

1.	Copper nitrate	4 oz.
	Ammonium chloride	4 "
	Calcium chloride	4 "
	Water	1 gal.
2.	Acetic acid	½ gal.
	Ammonium chloride	20 oz.
	Sodium chloride	7 "
	Cream of tartar	7 "
	Copper acetate	7 "
	Water	½ gal.

These can be stippled on brass or copper but the green color effect will vary according to the alloy of the basis metal. The use of sodium salts impart a yellowish color to the green; the ammonium salts a bluish cast.

The articles, if made of copper or copper plated, can be colored black in a

sulfide solution and then stippled. This will give a background of a dark color that cannot be obtained on the clear copper.

If, when the article is partly stippled and the antique green color begins to develop, the article is immersed in clean boiling water several different color effects may be obtained.

By incorporating some dry colors, e.g. light and dark chrome green; burnt and raw sienna; burnt and raw umber; ivory drop white; drop black or Indian red; a vast number of effects can be obtained. By this method almost any of the colors found on ancient bronzes can be reproduced.

After lacquering these finishes, the gloss of the lacquer can be removed by brushing with paraffin, beeswax or carnauba wax on a goats-hair wheel, operated at about 750 r.p.m.

Tiffany Green

Copper sulfate	8 oz.
Ammonium chloride	4 "
Sodium chloride	4 "
Zinc chloride	1 "
Acetic acid	2 "
Water	1 gal.

The addition of 1 oz. of glycerine will prevent the green from drying too fast and produce a more even color. The articles are immersed in this solution and, if the color is not uniform, the immersions are repeated as often as desired, allowing the work to dry thoroughly between immersions.

ENGLISH BRONZE

This finish is also known as "Old English" and "English Brass" finish. Two solutions are required.

Solution No. 1:

Liver of sulfur or any polysulfide	½ oz.
Water	1 gal.

Solution No. 2:

Copper sulfate	2 oz.
Water	1 gal.

After cleaning thoroughly, the work is dipped in solution No. 1 and then into solution No. 2, without rinsing between dips. The articles are then rinsed in clean cold water and the dipping operations are repeated until the desired shade is obtained.

For an even finish, the articles are scratch brushed dry and dipped once again, in solution No. 1 alone for light shades and in No. 1 and No. 2 for dark shades. They are then scratch brushed dry and lacquered.

SILVER

The finish used on silver is generally a gunmetal shade, known as "French Gray". This is produced by coloring the articles black and then relieving with pumice and water or with greaseless compound. The dark color is left in the recesses while the highlights are given a semi-gloss finish.

The black color may be produced with a weak solution of any polysulfide such as red ammonium sulfide, liver of sulfur, etc., used hot and preferably with the addition of ammonia, where a darker color is desired. A concentration of 1-2

oz./gal. is satisfactory. This process gives an adherent finish, permitting shading from a deep grayish-black to a lighter color and finally to the silver base. It may be applied with a brush, instead of by immersion, if only a portion of the article is to be "oxidized," less silver being removed in this way.

Another solution which may be used is tellurium chloride. This solution is prepared by dissolving tellurium dioxide in hydrochloric acid and diluting with water. Although it can be used for immersion purposes, it is generally employed with a brush for filling in the recesses in ornamented articles, such as borders on holloware, for example. It is used hot, and the article should be heated if possible.

Arsenic deposits have a color almost identical with silver sulfide and are being employed on jewelry and other items which do not come in contact with food. A formula for such solution appears in the section on Plating Solutions. Since no silver is used in the formation of the color, a thinner deposit may be employed, thus conserving the precious metal.

Silver flatware, which is often cleaned electrolytically, in the home and restaurant, in contact with a piece of aluminum or zinc, cannot be antiqued with sulfides, since the sulfide would be reduced to metallic silver during the cleaning process. Black nickel deposits are sometimes employed for this application.

COLORING ANODIZED ALUMINUM

By Edward Washburn

McKinney Mfg. Co., Coraopolis, Pa.

The coloring of anodized films is comparatively simple. Matching colors, however, requires skill. The final color is strongly influenced by the type of alloy used but all aluminum alloys that can be anodized can also be dyed and in general the lighter colors are more satisfactory on the purer forms of aluminum. Polishing, graining, etching, etc. should be done before anodizing, although the surface may be buffed after anodizing and dyeing in order to bring up a higher lustre. Before anodizing, high silicon alloys should be dipped *white* in a mixture of 90% nitric and 10% hydrofluoric acids followed by a dip in a mixture of 75% nitric and 25% sulfuric acids, if necessary.

SULFURIC ACID FILMS

Anodized films produced in the sulfuric acid electrolyte are generally easiest to color since they are thicker and more porous than films produced in the chromic acid bath and, as a result, have more affinity for the dyes. A solution containing about 15-25% sulfuric acid by weight is used and the temperature maintained at 60-80°F., employing a lead-, rubber- or brick-lined tank and lead cooling coils. The fumes should be exhausted during anodizing. Voltages range from 12 to 18 volts with a current density of approximately 8 to 20 amp./sq. ft. High silicon alloys give better films for coloring at lower voltages. Alloys of this type have been anodized at 6 volts, producing films in one hour which colored very well indeed.*

*Anodizing in a solution of sulfuric acid is a patented process of the Aluminum Co. of America, Pittsburgh, Pa.

CHROMIC ACID FILMS

The conditions of chromic acid anodizing are somewhat more critical. The usual 5-10% chromic acid baths are used but at higher temperatures and for longer anodizing periods. Anodizing at 100°F. produces a very good surface for coloring, except for high silicon alloys, in which case the temperature should not exceed 80-85°F. If the temperature is allowed to rise above about 120°F. the films tend to become powdery and less resistant to salt spray corrosion. Anodizing time has been variously reported from 30 to 120 minutes, using 40 volts except for high silicon alloys, in which case the voltage is dropped to 30. In a properly operated bath, 60 minutes produces a very good film for subsequent coloring.

The pH is maintained at 0.8 to 0.9 (1.25-1.5 for high silicon alloys gives better results). Batch type operation, in which a new solution is used until gradually exhausted, and then discarded, is an extremely unsatisfactory practice since the amount of built up impurities varies from beginning to end and has a decided effect on the darkness of the anodized film. Matching the colors produced on such films is a very difficult procedure. Uniform results are obtained by control of the bath according to the procedure given in the preceding section on ANODIZING ALUMINUM, namely periodic withdrawals of portions of the solution and replacement with chromic acid in amounts sufficient to adjust the pH to the proper value, maintaining the total chromic acid content at not more than 10%.

COLORING

After anodizing, articles are thoroughly rinsed in cold water. Hot water should not be used as it tends to seal the pores, impeding absorption of the dyes. The coloring processes consist of three main steps, all three being ordinarily used. They are:

1. Dyeing
2. Setting in Nickel Acetate
3. Sealing

Matching of colors requires that the original anodized films be of uniform thickness, porosity and color, as described above.

DYE BATHS

Color matching from day to day is accomplished by periodic additions of the dye and adjustment of pH to obtain a match on a test sample. Concentrations usually vary between 1 and 5 g./L. of dye. The addition of *small* amounts of acetic acid is employed in nearly all dye baths. The actual amount of acetic acid is best determined in production by gradually adding it until the desired evenness and intensity are obtained. It is not usually necessary to add more than 1 cc./L. When the correct amount of acid is determined, the pH should be checked electrometrically (quinhydrone or glass electrode) and maintained at that value thereafter. The pH will generally be between 4 and 8. Coloring time is from 15 to 30 minutes, no further change in color being produced by longer immersion in most cases. Containers are preferably Monel, but glass, enameled steel, and in some cases plain steel, may be used.

Some of the dyes and their average conditions which have been recommended are listed below. Varying the concentrations changes the shades. Combinations of dyes in different proportions will give unlimited range of tints.

Complete METAL CLEANING *Service*

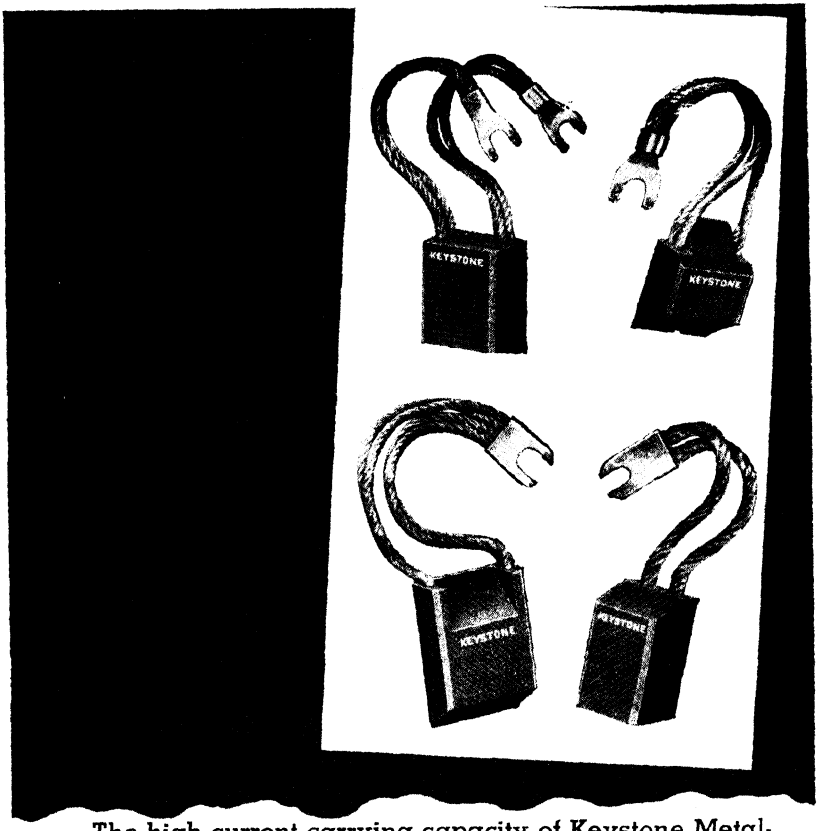
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KEMTEX COMPOUNDS

The trade name applied to a complete list of products for any industrial cleaning purpose. Materials for cleaning prior to plating, aluminum processing, chemical black processes, treatment for magnesium and stripping compounds for paints, enamels and phenolics.

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Dye	Conc. g./L.	cc. Acetic added	Time min.	Temp. °F.	Set
Red Dyes					
Azorubine Extra	5	1	30	180	Yes
Polar Red G-conc.	5	1	30	180	Yes
Guinea Red GL	5	1	30	180	Yes
Direct Fast Red 8BLA conc.	1	None	15	140	Yes
Neolan Red BRE	5	2	180	185	Yes
Cloth Red 2B Extra Conc.	3	1	30	180	Yes
Acid Orange GG Crystals	8	1	30	180	Yes
Erie Chrome Orange R	5	1	30	190	Yes
Yellow Dyes					
Quinoline Yellow P	1	1	30	190	Yes
Solatine Yellow FF conc.	5	None	30	180	Yes
Polar Yellow 5GN conc.	5	1	30	190	Yes
Wool Yellow Extra	5	1	30	200	Yes
Aluminum Yellow A	5	1	30	200	
Blue, Black and Brown Dyes					
Pontamine Fast Turquoise Blue 8GL	5	1	30	180	Yes
Aluminum Blue A	5	None	30	150	Yes
Erie Fast Cyanine S conc.	5	1	30	190	Yes
Niagara Sky 6B	5	1	30	180	Yes
Niagara Direct Blue RW	1	1	30	180	Yes
Nigrosine J Crystals	7	1	60	190	No
Nigrosine JB	5	None	30	180	No
Neutral Brown RX	5	1	30	190	Yes
Green Dyes					
Naphthol Green B	5	2	60	200	Yes
Direct Erie Green Gy	5	None	30	200	Yes
Pontamine Fast Turquoise Blue 8GL—1 part and Polar Yellow 5GN conc.—2 parts mixed together	5	1	15	190	Yes
Pontamine Fast Turquoise Blue 8GL—1 part and Quinoline Yellow P Extra conc.—2 parts mixed	5	1	60	180	Yes
Diphenyl Fast Blue-Green BL	5	1	30	190	Yes
Naphthaline Green V Extra conc.	5	1	30	210	Boil
Palatine Green BLNA conc. CF	5	2	30	210	Yes

It is also very interesting to note that anyone desiring to explore the possibilities of colored anodizing may get beautiful results with the common cloth dyes such as the Tintex and Diamond dyes. It is necessary to use a higher concentration of these dyes, such as 25 g./L., because they are not as strong as some of the dyes listed above. Acetic acid in the usual small quantities should be used. Other regular rules of procedure prevail.

SETTING

After dyeing, the articles may be set in a nickel acetate solution having a

concentration of 5 g./L. and maintained at a pH of 5 and a temperature of 200°F. or higher. Immersion for about 15 minutes sets the dye and increases their water fastness.

SEALING

The water fastness of most colors is further improved by immersing in boiling water for 15 to 30 minutes. Bleeding of the dye may occur at first but at the end of about 30 minutes there is seldom any further loss of color.

After dyeing, if the articles are placed in a drying oven at a temperature of 180-190°F. *before* setting or sealing, there is less bleeding and the original color from the dye bath is more readily retained. A cabinet heated with steam coils or radiators is satisfactory for this purpose.

Potassium and sodium dichromate are often used for sealing films produced in sulfuric acid anodizing baths but a yellow color is imparted to the finish.

METALLIZING NON-CONDUCTORS

SILVERING OF PLASTICS

By Harold Narcus

Technical Director, Electrochemical Industries, Inc., Worcester, Mass.

The demand for commercial procedures for the electrodeposition of metals on plastics, which have become established upon their merits in widely diversified fields, is increasing daily. The deposition of these various metals on plastics has opened up a large new field in electrical engineering and electronics in addition to the costume and novelty jewelry industries which, at the present time, are utilizing the process with excellent results. Such plated plastics combine all the inherent advantages of the plastic and the required properties of the outer electrodeposited metal. The net result is a combination of a non-conductor and a conductor which promises further wide use in future radio, television and electronic activities.

By plating on plastics, the undesirable properties of the plastics, such as absorption of oils, solvents and moisture, which may cause swelling or distortion of the basis organic material, are eliminated by proper choice and thickness of the outer layer of metal. The weatherability of the plastic is greatly increased. Experiments[1] show a marked increase in tensile, impact and flexural strength for many synthetic resins when plated as compared with values for the same resins unplated. An appreciable increase in resistance to distortion from heat and a decrease in percentage of water absorption are obtained by complete envelopment of the plastic in a suitable metallic coating. However, the most important advantage of plating on plastics is the greater corrosion resistance of a metallic deposit when it is applied to a plastic base than to the usual metallic base since there are no galvanic couples with a basis metal.

Advantages of Chemical Reduction Method

To render the plastic surface conductive, the methods using fine metal powders in a lacquer or varnish medium, metal spraying, cathode sputtering and metal evaporation have proven successful in specialized cases but were found inadequate for the majority of production methods in industry. On the other hand,

the chemical reduction method described in this section is best adapted to an economical productive set-up.

The main advantages of the chemical reduction method, especially the procedure using precipitated silver films, are (1) the silver film, in forming, covers the entire surface of the plastic regardless of its size or shape so that proper contact is immediately established over the entire surface; hence, instant plating takes place and the same thickness of metal results at every point on the surface; (2) the thickness of the very thin silver film can be controlled so as to permit subsequent electrodeposition of an outer metal in accurately determined magnitudes; (3) the fine details of certain specially-designed plastic parts are exactly reproduced or maintained, and (4) costly equipment and special techniques are not required.

Details of Chemical Reduction Method

There are numerous methods described in both the trade journals and patent literature[2, 9] for producing a conductive film on plastics for subsequent electrodeposition. In general, they accomplish the same result, namely, chemical reduction on a properly prepared plastic surface producing a highly conductive and reasonably adherent metallic film. A typical example includes use of an ammoniacal silver nitrate solution with a suitable organic reducing agent followed by a deposit of copper, silver or lead as an intermediate layer and, finally, a top layer of the desired metal such as chromium, nickel, gold, silver, cadmium, zinc or iron.

The preparatory treatments of the plastic surface are the governing factors which make for the success or failure of any process for "silvering" plastics since the chemical structure of the plastic determines the procedure to be employed for the application of the conductive film. The different types of plastic substances do not receive the same preparatory treatment. Some methods are applicable to one type of plastic while for another type they may be harmful and cause attack, excessive swelling, or even disintegration. The differences lie in the "de-glazing" of the hard plastic surface, its cleaning and so-called "sensitizing" (making the plastic surface more receptive to the conductive film to be formed thereon).

The following examples may be cited. Plastics such as the phenolics, ureas, cellulose nitrates, styrenes and methyl methacrylates are given a slight "de-glazing" operation before cleaning and "sensitizing" while cellulose acetates undergo a "priming" operation prior to "sensitizing" before the surface is highly receptive to a continuous, adherent silver film. Rubber compounds are prepared for metallizing[10] by immersion in benzol or acetone for roughening after which they are cleaned in a mild cleaner and "sensitized." Casein plastics use water as the swelling agent or "roughener" prior to their preparation to receive the silver film.[11] Urea-formaldehyde resins are roughened in 10% hydrochloric acid and treated in a 1% ammoniacal ferrous sulfate solution for 15 minutes at 30° C. They are then placed in a 2% copper sulfate solution for 3 minutes at 30° C.[12].

In general the formation of a suitably conductive and reasonably adherent silver film on a plastic surface involves the following steps:

(1) Slight roughening or "de-glazing" of the plastic surface.

(2) Cleaning the surface.

(3) "Sensitizing" the surface.

(4) Formation of the silver film by chemical reduction.

The electrodeposition of an intermediate layer of copper, silver or lead and the application of the desired outer layer of metal then follows.

Roughening—"De-glazing"

This procedure has two fundamental purposes: (1) to roughen the surface slightly or to remove glaze from the plastic article as it comes from the molding operation; and (2) to remove the "flash" or feather-edge from the molded piece. This operation is usually performed by (1) wet-tumbling in a mixture of #0½ pumice and water; (2) air-blasting with 220 mesh grain aluminum oxide, or (3) etching the part chemically. The surface preparation procedure used depends upon the size, shape and number of pieces to be treated and the chemical structure of the plastic. If the parts are small in size, the wet-tumbling procedure is employed, using tumbling barrels rotating at 40-50 revolutions per minute for one to five hours, depending upon the intricacy of the piece. Larger pieces are "de-glazed" with the aluminum oxide blast in the conventional type of blast cabinet.

Chemical roughening methods must be used with extreme care so that the etchant will not cause too severe an etching action. Plastics of the phenol-formaldehyde and urea-formaldehyde types as well as the cellulose esters can be treated in the usual acid bright dip solution (sulphuric-nitric-hydrochloric acid mixture) provided the excess acid is immediately rinsed away and the parts are immersed in a neutralizing solution such as a 10% sodium carbonate solution. Treatment for one to five minutes in a 5% to 10% sodium hydroxide solution may be used for etching plastics of the cellulose ester group; also organic reagents such as a 10% acetone solution.

Cleaning the Surface

The surface of the plastic must be freed from any greasy film, finger-marks or contamination following the roughening operation, otherwise a non-adherent and mottled silver film may result. Mild proprietary cleaners such as those employed for cleaning non-ferrous metals suffice in the majority of cases. Carbon tetrachloride or solutions of tri-sodium phosphate, or wetting agents, or mild caustic soda solutions may also be used. However, the chemical structure of the plastic material must be considered, in order to avoid chemical reaction between the cleaning agent and the plastic surface. Elevated temperatures may be used in cleaning provided these temperatures do not approach the distortion point of the plastic.

The plastic piece should be thoroughly rinsed in water after cleaning and not allowed to dry prior to the next step in the preparation of the plastic surface for receiving the subsequent silver film.

"Sensitizing"

The most important step in the preparation of the plastic surface to receive a continuous and adherent metallic film that is capable of carrying current is the "sensitizing" treatment.

The "sensitizing" treatment is best applied by immersion of the plastic part for one to two minutes in an aqueous solution of the agent. The reagent used must be thoroughly washed away with water, since incomplete removal results in a mottled, poorly adherent metallic silver film.

Freund[13] uses a solution of the following formulation:

Sulphuric acid (66° Bé)	100 cc.
Potassium dichromate	15 grams
Water	25-30 cc.

Weiss[14] suggests immersion for ½-1 hour in a mixture made up of the following chemicals:

Stannous sulfate	25-40 grams
Sulphuric acid (66° Bé)	5-20 cc.
Ethyl alcohol	150-250 cc.
Quinol	5-15 grams
Water	600-1000 cc.

Walker[15] recommends a solution of the following composition:

Stannous chloride	360 grams
Hydrochloric acid (C.P.)	216 cc.
Water	4000 cc.

Some prefer a more dilute stannous chloride solution represented by the formula:

Stannous chloride	10 grams
Hydrochloric acid (C.P.)	40 cc.
Water	1000 cc.

Formation of the Silver Film

The conductive silver film is produced on the properly cleaned and "sensitized" plastic surface by chemically reducing an ammoniacal solution of silver nitrate by means of a reducing agent. Bureau of Standards Circular #389 (1931)[16] summarizes three methods for producing silver films or mirrors on glass which work equally as well on organic plastics. However, in commercial applications, the following modified formulation, using formaldehyde (40%) as the reducing agent, is being employed with excellent results:

Ammoniacal Silver Nitrate Solution

Silver nitrate (C.P.)	60 grams
Distilled water	1000 cc.
Ammonium hydroxide (28%)	60 cc.

Reducing Solution

Formaldehyde (40% by volume)	65 cc.
Distilled water	1000 cc.

Although complete details for making up the ammoniacal nitrate solution are found in the previously mentioned governmental circular it would be well to review the procedure briefly since the amount of free ammonia in this silvering solution is most important. A relatively small excess of ammonia will prevent any deposition of silver on the plastic surface while too little ammonia will result in an excess of precipitated oxide which is undesirable.

The 60 grams of C.P. Silver Nitrate are dissolved in 1000 cc. *completely* and the concentrated ammonium hydroxide added with constant stirring. The precipitate first formed will redissolve when the entire amount of ammonium hydroxide is added. The resulting solution, after 60 cc. of concentrated ammonium hydroxide has been added will contain the proper amount of free ammonia for a suitable silvering solution. It must be stressed that only concentrated ammonium hydroxide (28%—Sp.Gr. 0.90) should be used in the above formulation. If the strength of the ammonium hydroxide has decreased upon standing a proportionate amount more must be used in the formula.

The reducing solution is made by adding 65 cc. of formaldehyde (40% by volume) to 1000 cc. of distilled water. Here again, if the strength of the formaldehyde has lessened a proportionate increase in the amount of formaldehyde must be employed.

The plastic parts, after being properly prepared, if in bulk, are "bonded" or "silvered" by placing them in an inclined rubber or synthetic lined container rotating at 4-5 revolutions per minute with enough water to completely cover the pieces. The container is best to possess ribs on its inner wall and bottom to insure against mass rolling of the parts which results in partial silvering. The proper volume of the freshly prepared ammoniacal silver nitrate solution is then added and an equal volume of the formaldehyde reducing solution. The quantity of the silvering solution required depends upon the total surface area of the work being "bonded" keeping in mind that the container's inner surfaces will also utilize some of the available silver. Larger pieces, not suitable for bulk handling as described are treated on racks or, in some cases, are sprayed with the silver and reducing solutions simultaneously hitting their surfaces through a specially-designed dual spray gun.

The "silvering" treatment continues until all the silver has been precipitated from the ammoniacal silver nitrate solution. This point may be determined by removing a sample of the "bonding" mixture and testing for precipitation of silver chloride with a 10% sodium chloride solution. The "bonding" operation usually requires 20 to 30 minutes to produce a silver film capable of carrying current to receive an electrodeposit of the intermediate layer of metal. A suitable silver film can be tested with an ohmmeter for conductivity or with a flashlight bulb connected in series with two test prods and a dry cell.

Following the "bonding" treatment the parts are rinsed thoroughly and allowed to dry preferably overnight or by a hot air blast.

The intermediate layer of metal is best applied from the conventional type of acid copper plating bath. This deposit, after bright-dipping, buffing or tumbling is then ready for the desired final coat of metal.

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BRONZING BABY SHOES

By G. B. Hogaboom, Jr. and Nathaniel Hall
Newark, N. J.

The first step in metallizing such articles is to tack down the laces, after which the leather is sealed by soaking in lacquer, shellac or wax. The last is generally employed, even though additional steps are required, because it is possible to shape the shoes while the wax is still soft. Paraffin or a mixture of paraffin and beeswax is used.

When the wax has set, two coats of shellac are applied by dipping, to prevent action of the lacquer thinner on the wax. Although the conducting film may be sprayed on the shellac surface directly, an intermediate coat of boiled linseed oil should be applied and allowed to dry, if the bronzing lacquer is to be brushed on, since the lacquer does not cover shellac well when brushed on. If linseed oil has been used, it is considered good practice to apply a coat of regular lacquer before bronzing, since the bronzing lacquer does not contain sufficient solids for good adhesion.

The bronzing mixture consists of the following:

Nitrocellulose lacquer	1 fl. oz.
Lacquer thinner	7 " "
Copper bronze powder	2 oz.

Only enough for immediate use should be prepared since the metal powder often causes the lacquer to jell. The lacquer should be low in gum (less than 5%) and should be added after the thinner and powder have been well mixed. If the copper powder is greasy, it should be washed with thinner before using. A satisfactory powder may be purchased from B. F. Drakenfeld & Co., Inc., 45 Park Place, New York City, under the name of "Extra Fine Platers Copper Powder." This powder is practically grease free and does not require washing with thinner.

When spraying, the gun is not held close enough to flood the surface but at a distance so that the film dries almost as soon as it is applied. The surface must be dull when dry. If glossy, it indicates that the copper is coated by a layer of lacquer which will prevent the passage of current. When completely covered (two coats are usually sufficient), the article is allowed to dry for 1-2 hours, fine wire is attached at a few points for distribution of current and the object is plated in the regular acid copper bath for about 2 hours at 20-25 amp./sq. ft. Most, if not all, of the object should be coated with copper deposit, by now, and the fine wires may be removed and replaced with a copper hook, attached at some point where the mark will not be noticeable, or by a single wire in contact with the copper deposit. The deposition of copper may now be continued as long as desired at from 10 amp./sq. ft. up, depending on the cleanliness of the solution and the agitation. Before plating, it is desirable to immerse the object in a cyanide silver solution having the same formula as the regular silver plating solution. This immersion coats the copper powder with silver and makes it a better conductor. Any spots which have been insulated by the lacquer show up in the original color, whereas those portions which are ready to conduct the current will take on an immersion silver deposit and will be of creamy color. The bad spots can then be touched up immediately, instead of showing up after copper plating.

After copper plating, the articles can be treated in the same manner as solid copper, to which oxidized, silver and gold finishes are applied, after polishing.

PLATING CALCULATIONS

DETERMINING SURFACE AREA OF STAMPINGS

By Nathaniel Hall and G. B. Hogaboom, Jr.

The accompanying charts may be used to determine the surface area of the great majority of stamped products of copper, brass and steel with an accuracy which is sufficient for all practical plating purposes and with a minimum of effort.

There are a few conditions which will affect the accuracy of these charts. If the stampings are of heavy gauge, and are either very small or have a large number of holes punched in them, the areas of the cut edges should be taken into account. Also if there is an appreciable reduction in the thickness of the sheet, such as is encountered in deep drawing, the final thickness of the stock will be less than the original gauge and the surface area will be greater by an amount depending on the amount of reduction.

To determine the surface area of an ordinary stamping, a sample, or a few samples if the articles are very light, is weighed up, and if the Brown and Sharpe gauge is not known, the thickness is measured with a micrometer caliper. The point on the horizontal scale corresponding to the weight of the sample in ounces or grams is followed vertically until the curve corresponding to the stock gauge or thickness is reached. A horizontal line drawn from this point will intersect the vertical axis at a point corresponding to the area of the sample.

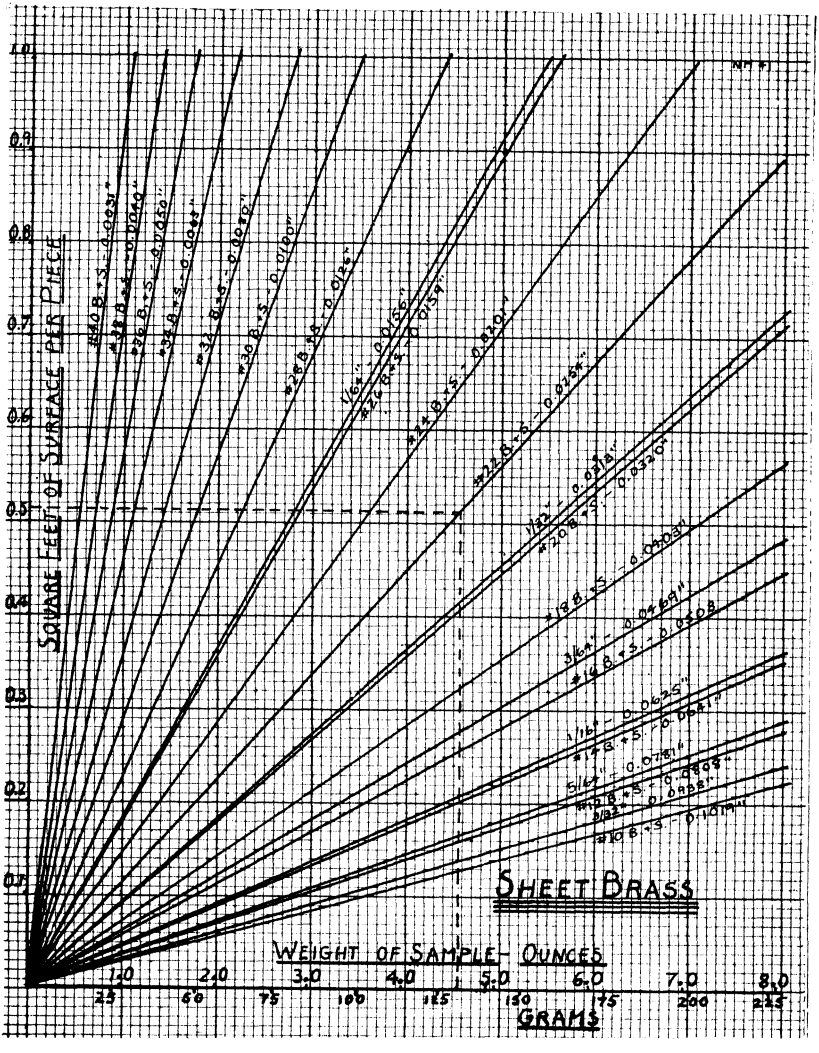
For example, if a brass stamping of #22 B. & S. stock weighs 4.6 ounces, a line is drawn vertically at this weight on Figure 1, which is for sheet brass, until it hits the 22 gauge curve (see dotted lines, Fig. 1). A horizontal line drawn from this point intersects the vertical axis at 0.514 square feet. If the samples are very light, a number of pieces are weighed together, in which case the surface area will be that for the sum of the pieces.

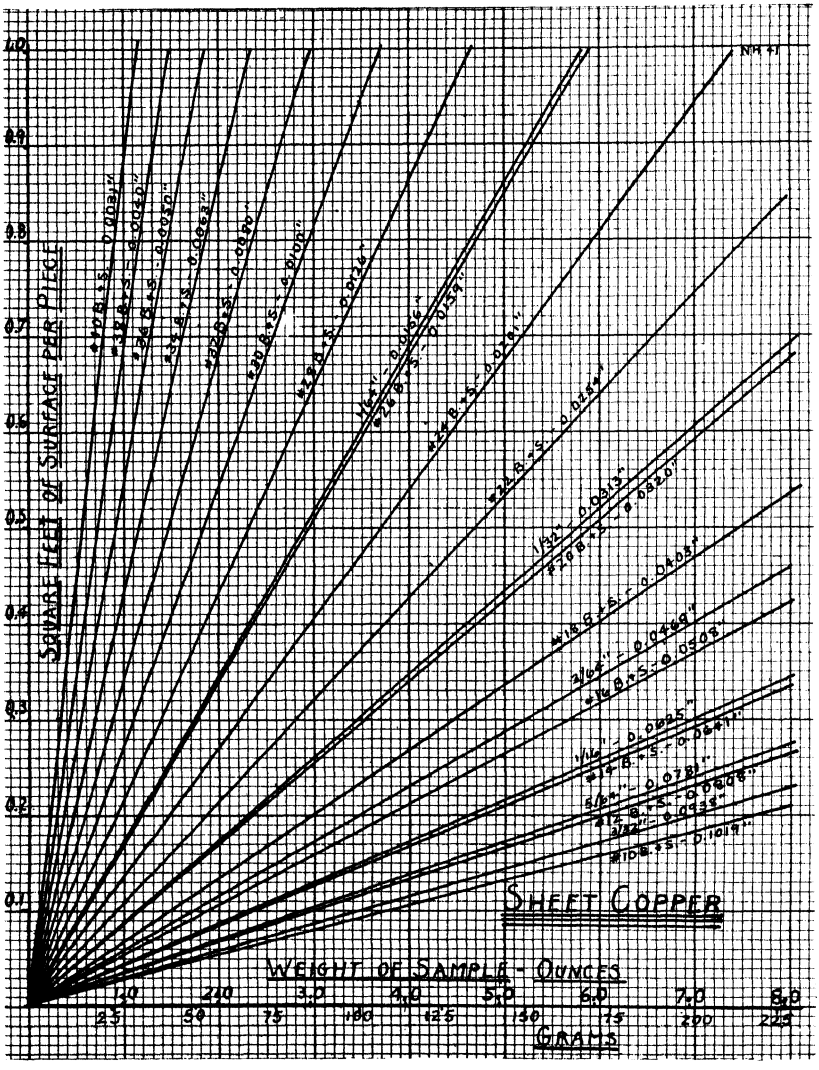
Occasionally it will be found that a large stamping of light gauge or a heavy stamping will not fit the scale. In such cases an appropriate fraction of the weight is used with the chart.

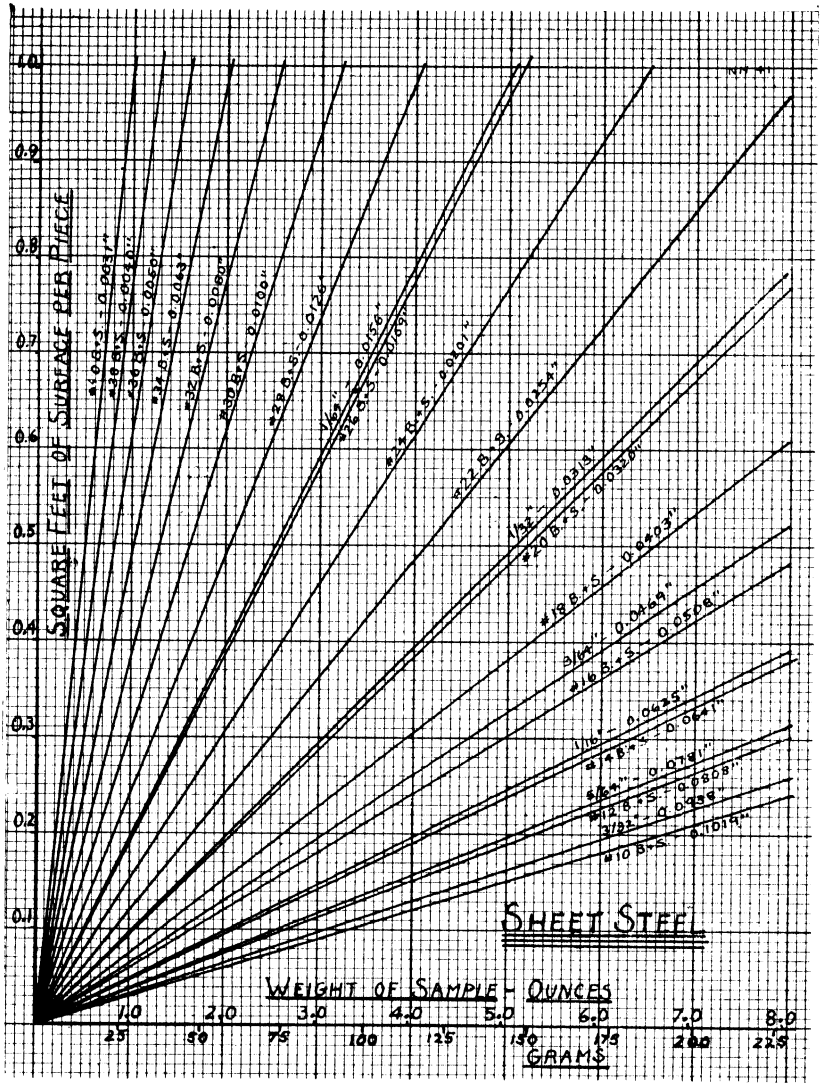
For example, a brass stamping of #30 B. & S. brass weighs 4.2 oz. We find that the 4.2 oz. line in Fig. 1 does not intersect the #30 B. & S. Curve. We therefore, look up the area of half this stamping which would weigh 2.1 oz. and find it to be 0.595 square feet. The whole stamping, therefore, has an area twice this or 1.19 square feet.

As another example, suppose a stamping of 1/16" brass stock weighs 23 oz. Since the scale goes only to 8 oz., we would look up an area corresponding to say one-tenth the stamping or 2.3 oz. and find it to be 0.103 square feet. The area of the stamping would, therefore, be ten times this or 1.03 square feet.

Charts such as these are much more satisfactory than the common practice of tracing outlines of the stamping on cross-section paper and adding up the squares, especially on stampings of complicated shape. The determination requires much less time and all that is necessary is a knowledge of the thickness of the metal and a small scale which will read in ounces or in grams. A postal scale is usually sufficiently accurate for this purpose and is available almost everywhere.


SQUARE FEET OF SURFACE PER PIECE
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
#40 B.+S. - 0.0031"
#38 B.+S. - 0.0040"
#36 B.+S. - 0.0050"
#34 B.+S. - 0.0063"
#32 B.+S. - 0.0080"
#30 B.+S. - 0.0100"
#28 B.+S. - 0.0126"
1/16" - 0.0156"
#26 B.+S. - 0.0159"
#24 B.+S. - 0.0201"
#22 B.+S. - 0.0253"
1/32" - 0.0312"
#20 B.+S. - 0.0320"
#18 B.+S. - 0.0403"
3/64" - 0.0469"
#16 B.+S. - 0.0508"
1/16" - 0.0625"
#14 B.+S. - 0.0641"
5/64" - 0.0781"
#12 B.+S. - 0.0808"
3/32" - 0.0938"
#10 B.+S. - 0.1019"
SHEET BRASS
WEIGHT OF SAMPLE - OUNCES
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
25
50
75
100
125
150
175
200
225
GRAMS


SQUARE INCH OF SURFACE PER PIECE
WEIGHT OF SAMPLE - OUNCES
GRAMS
SHEET COPPER
NR 41
0.0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1.0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
25
50
75
100
125
150
175
200
225



NOMOGRAPH ON THICKNESSES OF ELECTRODEPOSITS

By Arthur S. Covert

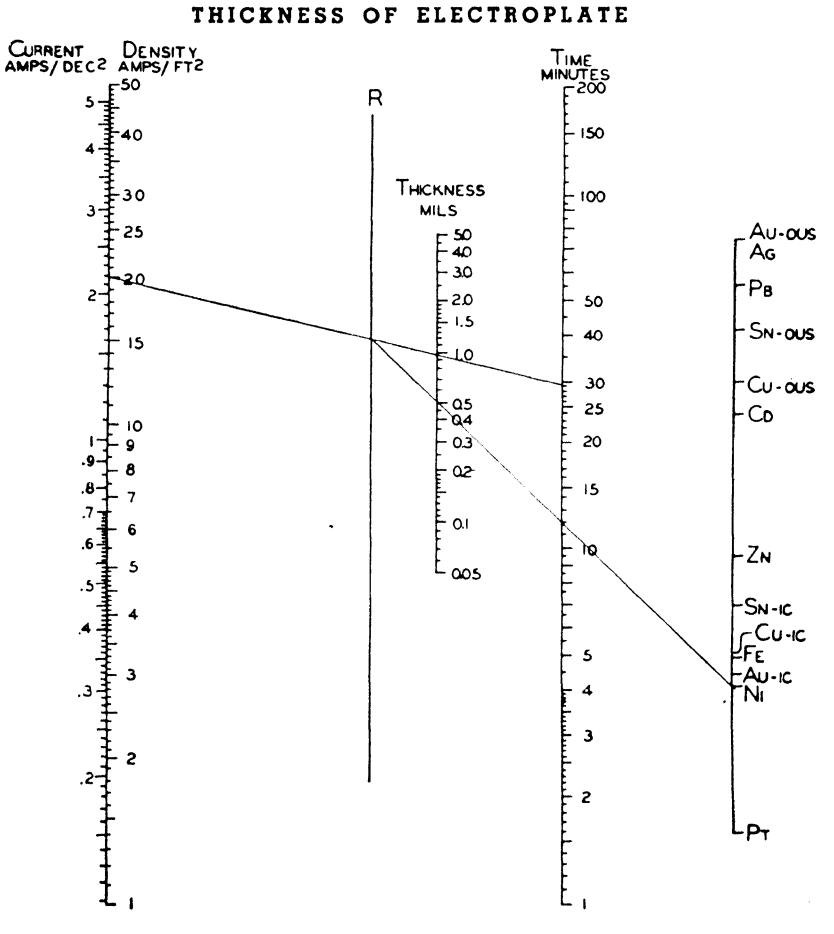
The nomograph on page 154 is designed to answer the questions most frequently met by the plater who wishes to deposit a given thickness of metal. Given the metal to be plated and the current density, it indicates the time required to

deposit a given thickness or conversely the thickness achieved in a known time.

For example, suppose it is desired to find the time necessary to produce a nickel plate 0.5 mils (0.0005 inches) thick at a current density of 20 amperes per square foot. To determine this from the chart, draw a line with a straight edge from the nickel point through the 0.5 mil point intersecting the reference line R. This intersection is connected to the 20 amperes per square foot point by another straight line, which when extended to the time scale gives the required time, in this case 29 minutes.

To calculate the amount of metal deposited in a given time, this procedure is reversed.

It must be noted that this nomograph is based on 100% current efficiencies, hence the actual plating times will be greater. Typical current efficiencies for various baths may be found on page 205. In the above example if the current efficiency were 95% the total plating time would be, 29 divided by 0.95 or 30.5 minutes.

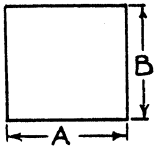


MEASURING SURFACE AREA

By Nathaniel Hall and G. B. Hogaboom, Jr.

In order to determine the time required to deposit a desired thickness of a given metal 4 factors must be known. The amperes may be read from the tank ammeter. The approximate efficiency can be found in the tables for the different plating solutions. The electrochemical equivalent, or, for practical purposes, the current density in amperes per square foot required to deposit a thickness of 0.001" per hour will also be found in the tables.

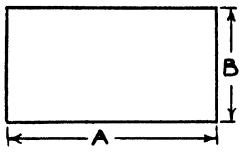
The most troublesome factor is the area of the surface to be plated, but, this determination may be simplified by breaking down complicated shapes into simple geometric figures, the areas of which are determined using the following formulas:



SQUARE:

Area = A × B

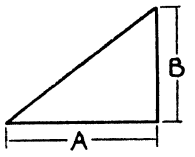
Example: Square 2" on a side.
Area = 2 × 2 = 4 sq. in.

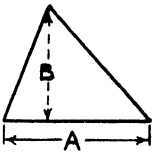


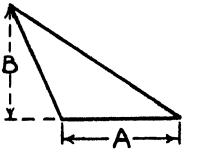
RECTANGLE:

Area = A × B

Example: Rectangle 3" by 2".
Area = 3 × 2 = 6 sq. in.

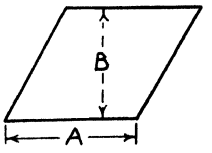






TRIANGLE:

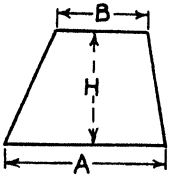
Area = ½ × base × altitude = ½ × A × B
Example: Triangle with base of 4" and height of 3".
Area = ½ × 4 × 3 = 6 sq. in.



PARALLELOGRAM:
(Opposite sides parallel)

Area = length of side × vertical distance to parallel side = A × B

Example: Parallelogram with one side of 3" and a distance of 2" between this side and the parallel side.
Area = 3 × 2 = 6 sq. in.



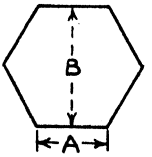
TRAPEZOID:

(One pair of opposite sides parallel)

Area = ½ sum of the parallel sides × vertical distance between them = ½ × (A + B) × H

Example: Trapezoid having parallel sides 3" and 2" long respectively, distance between them being 2".

Area = ½ × (3 + 2) × 2 = 5 sq. in.



HEXAGON:

(Six equal sides, all angles equal)

Area = 3/2 × length of one side × distance between any two parallel sides = 3/2 × A × B

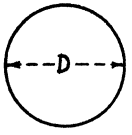
Example: Hexagon 1.16" on a side and 2" between flats.

Area = 3/2 × 1.16 × 2 = 3.48 sq. in.

Area = 0.87 × square of distance between any two parallel sides = 0.87 × B^2 = 0.87 × B × B

Example: Same as above.

Area = 0.87 × 2 × 2 = 3.48 sq. in.

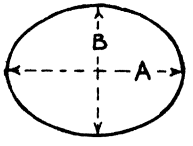


CIRCLE:

Area = 0.785 × square of the diameter = 0.785 × D^2 = 0.785 × D × D

Example: Circle 2" in diameter.

Area = 0.785 × $(2)^2$ = 0.785 × 2 × 2 = 3.14 sq. in.

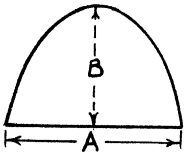


ELLIPSE:

Area = 0.785 × long diameter × short diameter = 0.785 × A × B

Example: Ellipse 3" × 2".

Area = 0.785 × 3 × 2 = 4.71 sq. in.

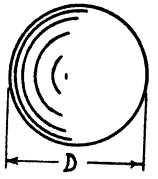


PARABOLA:

Area = 2/3 base × height = 2/3 × A × B

Example: Parabola with 3" base and height of 4".

Area = 2/3 × 3 × 4 = 8 sq. in.

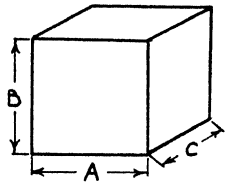


SPHERE:

Area = 3.14 × square of diameter = 3.14 × D^2 = 3.14 × D × D

Example: Sphere 2" in diameter.

Area = 3.14 × $(2)^2$ = 3.14 × 2 × 2 = 12.56 sq. in.



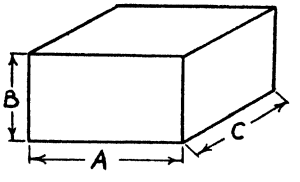
CUBE:

(All sides equal)

Area = 6 × square of one side = $6A^2$ = 6 × A × A

Example: Cube 2" on a side.

Area = 6 × $(2)^2$ = 6 × 2 × 2 = 24 sq. in.



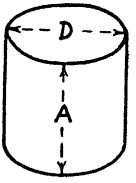
RECTANGULAR PARALLELOPIPED:

(Opposite sides parallel)

Area = 2 × (AB + BC + CA)

Example: Block having sides 2", 3" and 4" long respectively.

Area = 2 × (2 × 3 + 3 × 4 + 4 × 2) = 2 × (6 + 12 + 8) = 52 sq. in.



CYLINDER:

1. HOLLOW:

Area = Circumference × length = 3.14 × D × A

Example: Tube 2" in diameter and 6" long.

Area = 3.14 × 2 × 6 = 37.7 sq. in.

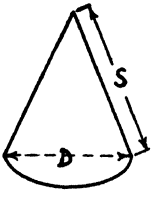
(Note: If outside and inside areas are to be determined the average of the inside and outside diameters should be used in the formula and the result doubled.)

2. SOLID:

Area = Circumference × length plus twice cross-sectional area = 3.14 × D × A + 1.57 × D × D

Example: Round bar 2" in diameter and 6" long.

Area = 3.14 × 2 × 6 + 1.57 × 2 × 2 = 37.7 + 6.28 = 44.0 sq. in.



CONE:

1. HOLLOW:

Area = ½ × circumference of base × slant height = ½ × 3.14 × D × S = 1.57 × D × S

Example: Cone 2" in diameter at base and 3" slant height.

Area = 1.57 × 2 × 3 = 9.42 sq. in.

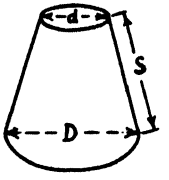
(Note: If outside and inside areas are to be determined the average of the outside and inside diameters and of the outside and inside slant heights should be used in the formula and the result doubled.)

2. SOLID:

Area = ½ × circumference of base × slant height + area of base = 1.57 × D × S + 0.785 × D × D

Example: Same as above.

Area = 1.57 × 2 × 3 + 0.785 × 2 × 2 = 9.42 + 3.14 = 12.56 sq. in.



FRUSTRUM OF CONE:

1. HOLLOW:

Area = ½ sum of circumferences of bases × slant height = 1.57 × (D + d) × S

Example: Tapered tube, 2" diameter at small end, 3" diameter at large end and 3" slant height.
Area = 1.57 × (3 + 2) × 3 = 23.6 sq. in.

(Note: If outside and inside areas are to be determined the average of the outside and inside diameters should be used in the formula and the result doubled.)

2. SOLID:

Area = ½ sum of circumferences of bases × slant height + sum of areas of bases
= 1.57 × (D + d) × S + 0.785 × (D^2 + d^2)
= 1.57 × (D + d) × S + 0.785 × (D × D + d × d)

Example: Tapered bar, 2" diameter at small end, 3" diameter at large end and 3" slant height
Area = 1.57 × (3 + 2) × 3 + 0.785 × (3 × 3 + 2 × 2) = 23.6 + 10.2 = 33.8 sq. in.

IDENTIFICATION OF DEPOSITS

Lacquer coatings, when present, should be removed with thinner, after which the metal deposit is scratch brushed lightly or scrubbed with a paste of magnesia powder and water and rinsed well. The deposit is then treated with a solution of equal parts of nitric acid and water for about two minutes.

PLATING NOT ATTACKED BY HNO_3: Indicates aluminum, chromium, gold or platinum group metals (palladium, platinum, rhodium).

1. *Gold:* A colored deposit unattacked by nitric acid would be gold or a gold alloy.
2. *Aluminum:* Treat the deposit with 10% caustic soda solution. If it is attacked, aluminum is indicated.
3. *Chromium:* Treat the deposit with concentrated hydrochloric acid. Development of a green solution indicates chromium.
4. *Platinum Group Metals:* White deposits which are unaffected by either nitric acid or hydrochloric acid are in this group.

PLATING ATTACKED BY HNO_3:

A. Blue or Green Solution: Indicates chromium, copper, copper alloys or nickel.

1. *Copper*: Evaporate the solution to dryness. Dissolve the residue in 1 cc. of 20% by volume solution of sulfuric acid, then dilute to 100 cc. Immerse an iron nail in this solution for 4 hours. A red coating indicates copper or a copper alloy.
2. *Nickel:* Make the solution alkaline to litmus with concentrated ammonia and add 1 cc. of a 1% solution of dimethylglyoxime in 95% ethyl alcohol. A flocculent reddish or pink precipitate indicates nickel.
3. *Chromium:* If the tests for nickel and copper are negative, the deposit is chromium.

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Bulletin A-4455—"Zin-O-Lyte" Bright Zinc Plating Process

Bulletin A-4456—"Moly-Black" Molybdenum-Nickel Electroplating Process.

Bulletin A-4119—"Cadalyte" Cadmium Plating

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B. Cloudy White Solution: Indicates tin.

1. *Tin:* Dissolve another sample in concentrated hydrochloric acid and add solid cacotheline. A reddish-violet color will confirm the presence of tin.

C. Colorless Solution: Indicates cadmium, lead, silver or zinc.

1. *Zinc:* Make the solution alkaline to litmus with concentrated ammonia. Then add 10% sodium sulfide solution. A white precipitate indicates zinc.
2. *Cadmium:* Make the solution alkaline to litmus with concentrated ammonia. Then add 10% sodium sulfide solution. A yellow precipitate indicates cadmium.
3. *Silver:* Add 10% caustic soda solution until the solution is alkaline to litmus. A brownish-black precipitate indicates silver.
4. *Lead:* If zinc and cadmium are absent, add 10% caustic soda solution until the solution is alkaline to litmus. A white precipitate indicates lead.

In making tests for deposits by the above procedure it is important to be certain that the attack noted is attack of the deposit and not of the basis metal. For example, in the case of gold deposits on brass a blue solution will be obtained in a few seconds if the deposit is thin. Insoluble flakes of gold, however, will be seen to be present in the resultant solution.

ANALYSIS AND TESTING

ANALYZING PLATING SOLUTIONS

By Nathaniel Hall and G. B. Hogaboom, Jr.

In the following pages are given the methods of analyzing solutions. It is important to note that in connection with these methods, has come a growing movement to standardize the laboratory technique and equipment required. As a result, "testing sets" have been made available to the industry by a number of manufacturers, the use of which has simplified the operations to such an extent that analyses can now be made quickly and accurately by men who are not trained chemists. Any plater can, in a short time, learn to operate the apparatus and perform the operations. No complicated mathematical computations are required. Sets are now available for analyzing all of the standard solutions used in plating plants; brass and bronze, cadmium, chromium, acid copper, cyanide copper, acid gold, cyanide gold, nickel, silver, acid zinc and cyanide zinc, etc.

The standard solutions used in the following methods of analysis can be purchased made up ready to use. (Write *Metal Finishing* for names of suppliers.)

Taking Sample of Plating Bath

1. Bring the solution up to the correct tank level. If this is not done the analysis will not be correct.
2. Stir the plating solution thoroughly.
3. Take a 100 cc. sample. A large sample is more likely to be representative of the plating solution than a small one. It is a good idea to take the sample by sticking a long glass tube into the plating solution, allowing it to fill and then, holding your finger over the end of the tube, withdraw a column of liquid from the tank and allow it to drain into a dry flask by

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removing your finger from the end of the tube. If this method is used, a cross section sample of the plating solution will be obtained which will be more indicative of the bath itself.

4. Filter the plating solution sample before starting the analysis to insure removal of foreign matter.

I. ARSENIC SOLUTION

A. Free Cyanide:

1. Pipette a 10 cc. sample into a 250 cc. erlenmeyer flask.
2. Add 100 cc. of distilled water and 5 cc. of 10% potassium iodide (KI) solution.
3. Titrate with standard 0.1 N silver nitrate ($AgNO_3$) solution until a faint yellowish turbidity persists.

The standard $AgNO_3$ solution is prepared by weighing out exactly 17.00 g. of the C. P. salt and dissolving in distilled water, after which it is diluted to exactly 1 liter in a volumetric flask.

The **SODIUM CYANIDE** (NaCN) in oz./gal. is equal to: 0.131 x cc. of 0.1 N $AgNO_3$ required for sample of solution.

B. Carbonates:

1. Proceed exactly as in III-C on page 171.

C. Metallic Arsenic:

1. Pipette a 5 cc. sample into a 250 cc. erlenmeyer flask.
2. Add 100 cc. of distilled water and a few drops of 2% phenolphthalein in alcohol solution.
3. Add 10% hydrochloric acid (HCl) solution cautiously until the pink color disappears.
4. Add 2-3 grams of sodium bicarbonate ($NaHCO_3$) and 2 cc. of starch indicator, prepared as in II-C-8 on page 165 and titrate with standard 0.25 N iodine solution until a permanent blue color appears.

The standard iodine solution is prepared as follows:

60 g. of potassium iodide (KI) are dissolved in 200 cc. of distilled water. 32 grams of pure iodine (I_2) are added and the whole diluted to 1 liter after the I_2 has dissolved. Exactly 0.33 g. of C. P. arsenic trioxide (As_2O_3) containing 0.25 g. of arsenic is weighed out and dissolved in a 250 cc. erlenmeyer flask with 1 cc. of concentrated HCl. To this is added 100 cc. of distilled water and then 2-3 g. of $NaHCO_3$ and 2 cc. of starch indicator. The solution is now titrated as above with the standard iodine solution.

The **METALLIC ARSENIC** (As) in oz./gal. equals:

$$\left\{ \frac{6.68}{\text{cc. iodine required for 0.33 g. arsenic trioxide}} \right\} \times \text{cc. of iodine required for sample of solution.}$$

The figures inside the brackets represent the factor for the standard iodine.

D. Sodium Hydroxide

1. Pipette a 5 cc. sample into a 250 cc. erlenmeyer flask.
2. Add 10 cc. water and 8 drops of LaMotte Sulfo-Orange Indicator.
3. Titrate with standard 1.0 N hydrochloric acid (HCl) solution prepared as in III-C-7 on page 171 until the color changes from orange to yellow.



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The **SODIUM HYDROXIDE** (NaOH) in oz./gal. equals:

[1.07 × Normality of HCl] × cc. of HCl required for sample of solution.

The figures inside the brackets represent the factor for the standard hydrochloric acid.

II. BRASS AND BRONZE SOLUTIONS

A. Free Cyanide

1. Proceed as in I-A on page 163.

Note: The results in brass and bronze solutions are consistent only if exactly the same procedure is used for each analysis since the method does not strictly determine free cyanide due to the presence of an unknown amount of sodium zincate.

B. Carbonates:

1. Proceed exactly as in III-C on page 171.

C. Metallic Copper

1. Pipette a 10 cc. sample into a 250 cc. erlenmeyer flask.
2. Add 5 cc. concentrated sulfuric acid (H_2SO_4) and ½ cc. of concentrated nitric acid (HNO_3) from a 10 cc. graduate **while shaking under a hood.** (This operation must be carried out under a hood because poisonous cyanide fumes are evolved). Boil until dense white sulfur trioxide (SO_3) fumes are given off. (If all the nitric acid is not removed, subsequent titration will be difficult.) If the solution contains Rochelle salts, use 10 cc. of H_2SO_4 and as soon as the SO_3 fumes are given off, cool slightly, add 2 cc. of HNO_3 and boil to fuming in order to decompose the carbonaceous precipitate which will otherwise obscure the titration end-point later.
3. Cool and add 100 cc. of distilled water.
4. Add concentrated ammonium hydroxide (NH_4OH) until the solution is colored a dark blue and boil for 15 min.
5. Add 10 cc. of 30% acetic acid ($HC_2H_3O_2$) solution. The solution should be colored light blue.
6. Cool to room temperature, add 25 cc. of 20% potassium iodide (KI) solution and shake.
7. Titrate with standard sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$) solution until the brown color begins to turn yellow.
8. Add 2 cc. of 1% starch solution, prepared by making paste of 1 g. of starch with very little water and adding it to 100 cc. of boiling water, and continue titrating until the blue color disappears for about 1 minute.

The standard thiosulfate is prepared as follows: Dissolve 25 grams of $Na_2S_2O_3 \cdot 5H_2O$ in 1 liter of water. Weigh out exactly 0.20 gram of pure copper wire and dissolve in 1:1 nitric acid. Add 5 cc. concentrated H_2SO_4 and boil until SO_3 fumes are evolved. Cool, add 100 cc. of distilled water and add NH_4OH until the solution turns a deep blue color. Add 30% acetic acid solution until the color changes to a light blue or bluish green. Cool, add 25 cc. of 20% KI, shake and titrate with the thiosulfate solution as in II-C-7 and II-C-8 above.

The **METALLIC COPPER** (Cu) in oz./gal. equals:

$$\left\{ \frac{2.67}{\text{cc. thiosulfate required for 0.20 g. of copper}} \right\} \times \text{cc. of thiosulfate required for sample of solution.}$$

The figures inside the brackets represent the factor for the standard thiosulfate solution.



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D. Metallic Zinc:

1. Pipette a 10 cc. sample into a 250 cc. beaker.
2. Add 50 cc. of distilled water and heat almost to boiling.
3. Add, while stirring, 25 cc. of 15% sodium sulfide (Na_2S) solution and allow to settle. If the precipitate is dark, add some 3% sodium cyanide (NaCN) solution to whiten it.
4. Filter and wash with hot water containing a few drops of Na_2S solution. Discard the filtrate.
5. Transfer the precipitate and filter paper to the beaker in which the precipitation was made. Add 10 cc. of concentrated hydrochloric acid (HCl) and a few crystals of sodium sulfite ($Na_2SO_3 \cdot 7H_2O$).
6. Boil to expel the hydrogen sulfide (H_2S) gas, add 150 cc. of distilled water, heat almost to boiling and titrate with standard potassium ferrocyanide [$K_4Fe(CN)_6 \cdot 3H_2O$] solution using 5% uranium acetate [$UO_2(C_2H_3O_2)_2 \cdot 2H_2O$] solution as an outside indicator, until a drop of the solution added to a drop of the indicator on a porcelain plate results in a brown color.

The standard ferrocyanide is prepared as follows: 22 g. of the crystallized salt are dissolved in 1 liter of distilled water. Exactly 0.25 g. of pure granulated zinc (Zn) are dissolved in a mixture of 5 cc. HCl and 5 cc. of distilled water in a 250 cc. beaker. Add 150 cc. of distilled water and heat almost to boiling. Titrate with the standard ferrocyanide as above in II-D-6.

The **METALLIC ZINC** (Zn) in oz./gal. is equal to:

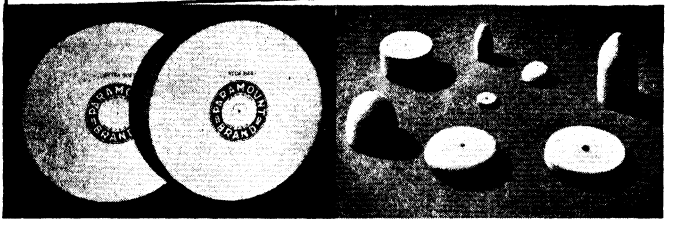
$$\left\{\frac{3.34}{\text{cc. of ferrocyanide sol. required for 0.25 g. zinc}}\right\} \times \text{cc. of ferrocyanide required for sample of solution.}$$

The figures inside the brackets represent the factor for the standard ferrocyanide solution.

E. Rochelle Salt:

1. Pipette a 5 cc. sample into a 250 cc. volumetric flask.
2. Add 100 cc. of distilled water, a pinhead of solid phenolphthalein powder and exactly 5 cc. of nitrobenzene.
3. Add 1% solution of sulfuric acid (H_2SO_4) dropwise until the pink color just disappears.
4. Add drop by drop, while shaking, 10% silver nitrate ($AgNO_3$) solution until the color of the precipitate changes from white to blue gray.
5. Shake for ½ minute to coagulate the precipitate and add 1 drop of $AgNO_3$ solution to the clear supernatant liquid to determine whether precipitation of the metal and cyanide is complete.
6. Bring the solution to the 250 cc. mark with distilled water, then add exactly 5 cc. more and shake thoroughly.
7. Allow to stand until the precipitate has settled and then draw out exactly 50 cc. of the clear liquid into a 500 cc. erlenmeyer flask.
8. Add 5 cc. of 20% H_2SO_4 solution, 5 grams of manganese sulfate ($MnSO_4 \cdot 4H_2O$) and 100 cc. of distilled water. Heat to about 160°F. and add slowly with agitation, exactly 20 cc. of standard 0.1 N potassium permanganate ($KMnO_4$) solution.
9. Allow the hot sample to stand for 5 minutes and cool to room temperature under running water.

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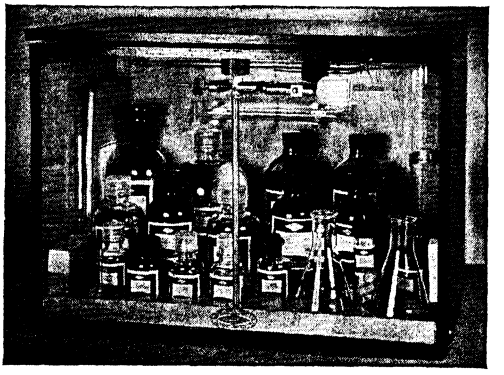
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10. Add 2 grams of solid potassium iodide (KI) and 2 cc. of 1% starch solution prepared as in II-C-8 on page 165, and shake the flask for a few seconds.
11. Titrate with 0.1 N sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$) solution until the blue color disappears for about 1 minute.

The standard thiosulfate is prepared as in II-C-8 on page 165. Its normality is equal to:

$$\frac{3.15}{\text{cc. thiosulfate required for 0.20 g. of copper}}$$

The standard permanganate is prepared as follows: Dissolve 3.2 grams of $KMnO_4$ in 1 liter of distilled water and let stand 24 hours. Pipette a 10 cc. sample into a 500 cc. erlenmeyer flask, add 100 cc. of distilled water, 5 cc. of 20% H_2SO_4 solution, 2 grams of potassium iodide and shake for a few seconds. Add 2 cc. of 1% starch indicator and titrate with the standard thiosulfate until the blue color disappears for about one minute:

The normality of the permanganate is equal to:

$$\frac{\text{cc. of thiosulfate required} \times \text{Normality of the thiosulfate}}{10}$$

The **ROCHELLE SALT** ($KNaC_4H_4O_6 \cdot 4H_2O$) in oz./gal. equals:
6.28 (20 × Normality of $KMnO_4$ — cc. $Na_2S_2O_3$ × Normality of $Na_2S_2O_3$)

III. CADMIUM SOLUTION

A. Metallic Cadmium:

1. Pipette a 5 cc. sample into a 400 cc. beaker.
2. Add 50 cc. of distilled water and warm.
3. Add, while stirring, 10 cc. of 15% sodium sulfide (Na_2S) solution and allow to settle.
4. Filter and wash with hot water containing a few drops of Na_2S solution.
5. Transfer the precipitate and filter paper to the beaker in which the precipitation was made, add 25 cc. of concentrated hydrochloric acid (HCl) and 25 cc. of distilled water and boil until the precipitate is dissolved.
6. Add 200 cc. of distilled water and then ammonium hydroxide (NH_4OH) until a piece of red litmus paper just turns blue.
7. Add concentrated HCl slowly until the litmus just turns red and then add 3 cc. in excess.
8. Heat almost to boiling and titrate with the standard ferrocyanide solution as in II-D-6 on page 167. The ferrocyanide is standardized against exactly 0.25 g. of metallic cadmium (Cd) just as against zinc, except that 10 g. of ammonium chloride (NH_4Cl) are added to the cadmium solution just before titrating.

The **METALLIC CADMIUM** (Cd) in oz./gal. is equal to:

$$\left\{\frac{6.68}{\text{cc. ferrocyanide solution required for 0.25 g. Cd}}\right\} \times \text{cc. ferrocyanide required for sample of solution.}$$

The figures inside the brackets represent the factor for the standard ferrocyanide solution.

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B. Total and Free Cyanide:

1. Pipette a 5 cc. sample into a 250 cc. erlenmeyer flask.
2. Add 100 cc. of distilled water, 5 cc. of 10% potassium iodide (KI) solution and 15 cc. of ammonium hydroxide (NH_4OH).
3. Titrate with the standard 0.1 N silver nitrate solution prepared as in I-A-3 on page 163, until a faint yellow turbidity is produced.

The **TOTAL SODIUM CYANIDE** (NaCN) in oz./gal. is equal to: 0.261 × $AgNO_3$ required for sample of solution.

The sodium cyanide combined with the metallic cadmium is equal to: 1.74 × oz./gal. metallic cadmium as determined in III-A-8 on page 169.

The **FREE SODIUM CYANIDE** (NaCN) in oz./gal. is equal to: Total sodium cyanide minus (1.74 × oz./gal. cadmium).

C. Carbonates:

1. Pipette a 10 cc. sample into a 250 cc. beaker.
2. Add 100 cc. of distilled water and warm.
3. Add 10% barium nitrate [$Ba(NO_3)_2$] solution, while stirring, until no more precipitate forms. Each cc. of the $Ba(NO_3)_2$ solution will precipitate an amount of carbonate equivalent to ½ oz./gal. sodium carbonate (Na_2CO_3). Allow to settle.
4. Filter, adding a few drops of $Ba(NO_3)_2$ solution to the first portion of filtrate to determine whether precipitation is complete. Wash with hot water.
5. Transfer the filter paper and precipitate to the beaker in which the precipitation was made.
6. Add 50 cc. of distilled water and a few drops of 0.2% methyl orange solution.
7. Titrate with approximately 1.0 N hydrochloric acid (HCl) until a permanent pink color is obtained.

The HCl is prepared by diluting 83 cc. of C.P. acid to 1 liter and standardizing as follows: A 5 cc. sample of the HCl is pipetted into a 250 cc. erlenmeyer flask and 50 cc. of distilled water are added. A 2% sodium hydroxide (NaOH) solution is added drop by drop until the solution is neutral to litmus. 1 cc. of 2% sodium chromate (Na_2CrO_4) solution is added and the solution is titrated with standard 0.1 N silver nitrate prepared as in I-A-3 on page 163, until a red precipitate just forms.

The **normality of the HCl** will be equal to:

$$\frac{\text{cc. of 0.1 N } AgNO_3 \text{ required}}{50}$$

Thus, if 46.5 cc. of $AgNO_3$ are required, the acid will be 46.5/50 or 0.93 N. As 1 cc. of 1.0 N HCl is equivalent to 0.0530 g. of Na_2CO_3, therefore:

The **SODIUM CARBONATE** (Na_2CO_3) in oz./gal. is equal to:

[0.706 × Normality of HCl] × cc. HCl required for sample.

The figures inside the parentheses represent the factor for the standard hydrochloric acid solution.

D. Sodium Hydroxide:

1. Pipette a 10 cc. sample into a 250 cc. erlenmeyer flask.
2. Add 10 cc. of distilled water, 1 gram of solid sodium cyanide and 8 drops of LaMotte Sulfo-Orange Indicator.
3. Titrate with standard 1.0 N hydrochloric acid (HCl) solution prepared as in III-C-7 (above) until the color changes from orange to yellow.

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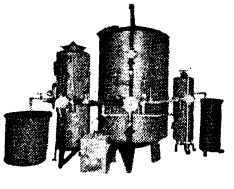
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[0.535 × Normality of HCl] × cc. of HCl required for sample of solution.

The figures inside the brackets represent the factor for the standard hydrochloric acid solution.

IV. CHROMIUM SOLUTION

A. Chromic Acid:

1. Determine the density of the solution with a Baume hydrometer.
2. Read the oz./gal. of chromic acid (CrO_3) corresponding to this density from the following table. The solution should be cooled to room temperature before testing.

Direct Conversion of Degrees Baume to Oz./gal. of Chromic Acid

Degrees Be.	Oz./gal.-CrO_3	Degrees Be.	Oz./gal.-CrO_3
1.50	2.1	19.00	29.0
2.00	2.8	19.50	29.8
2.50	3.4	20.00	30.6
3.00	4.1	20.50	31.5
3.50	4.8	21.00	32.4
4.00	5.5	21.50	33.3
4.50	6.2	22.00	34.2
5.00	6.8	22.50	35.1
5.50	7.5	23.00	36.0
6.00	8.2	23.50	37.1
6.50	8.9	24.00	38.2
7.00	9.7	24.50	39.1
7.50	10.4	25.00	40.0
8.00	11.1	25.50	40.9
8.50	11.9	26.00	41.9
9.00	12.6	26.50	42.9
9.50	13.4	27.00	44.0
10.00	14.2	27.50	45.0
10.50	15.0	28.00	46.0
11.00	15.8	28.50	47.1
11.50	16.5	29.00	48.2
12.00	17.3	29.50	49.2
12.50	18.2	30.00	50.2
13.00	19.1	30.50	51.5
13.50	19.8	31.00	52.7
14.00	20.4	31.50	54.0
14.50	21.2	32.00	55.2
15.00	22.0	32.50	56.3
15.50	22.9	33.00	57.5
16.00	23.7	33.50	58.7
16.50	24.5	34.00	60.0
17.00	25.4	34.50	61.2
17.50	26.3	35.00	62.3
18.00	27.2	35.50	63.5
18.50	28.1	36.00	64.8

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B. Trivalent Chromium:

1. Pipette a 10 cc. sample into a 400 cc. beaker and add 200 cc. of distilled water.
2. Add a slight excess of ammonium hydroxide (NH_4OH) and boil. Allow to settle and filter.
3. Wash precipitate with hot distilled water and dissolve with 25 cc. of 10% sulfuric acid (H_2SO_4) solution.
4. Add excess NH_4OH and 25 cc. of 10 volume hydrogen peroxide (H_2O_2). Boil for 15 min.
5. Filter to remove iron and add 20 cc. of 30% potassium acid sulfate ($KHSO_4$) solution to the filtrate. Boil for 15 min.
6. Add H_2SO_4 until neutral and then add 5 cc. excess.
7. Add about 2 g. of solid potassium iodide (KI) and after 5 min. titrate with standard 0.5 N sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$) until the brown color begins to lighten.
8. Add 2 cc. of 1% starch solution prepared by making a paste of 1 g. of starch with very little water and adding it to 100 cc. of boiling distilled water, and continue titrating until the blue color disappears for about 1 min.

The **standard thiosulfate** is prepared as follows: Dissolve 125 g. of sodium thiosulfate in water and dilute to one liter. Exactly 0.50 g. of C.P. potassium dichromate ($K_2Cr_2O_7$) containing 0.177 g. of chromium is weighed out and dissolved in 100 cc. of distilled water. 2 g. of KI and 5 cc. of sulfuric acid are added and after 5 min. the solution is titrated with thiosulfate and starch indicator as above. One cc. of 1.0 N thiosulfate is equivalent to 0.1733 g. of chromium.

Therefore the **normality of the thiosulfate** is equal to 0.177/(cc. of $Na_2S_2O_3$ required for 0.50 g. $K_2Cr_2O_7$ $\times$ 0.01733) equals 10.2/cc. $Na_2S_2O_3$ required for 0.50 g. $K_2Cr_2O_7$.

The **TRIVALENT CHROMIUM** (Cr) in oz./gal. is equal to:

$$\left\{\frac{2.36}{\text{cc. of thiosulfate soln. required for 0.50 g. } K_2Cr_2O_7}\right\} \times \text{cc. of thiosulfate required for sample of solution.}$$

The figures inside the brackets represent the factor for the standard sodium thiosulfate solution.

C. Sulfate:

1. Pipette a 10 cc. sample into a 400 cc. beaker and add 100 cc. distilled water.
2. Add 10 cc. of concentrated hydrochloric acid (HCl), 15 cc. of glacial acetic acid ($HC_2H_3O_2$) and 20 cc. of pure ethyl alcohol (C_2H_6OH) and boil for 30 minutes.
3. Add, while stirring, 15 cc. of 10% barium chloride ($BaCl_2 \cdot 2H_2O$) solution, boil for 1 min. and allow to settle in a warm place for 1 hour.
4. Filter, using a quantitative filter paper and wash.
5. Ignite the precipitate and filter paper in a weighed crucible. Cool in a desiccator and weigh.

The **SULFURIC ACID** (H_2SO_4) in oz./gal. is equal to: 5.64 $\times$ gain in weight of the crucible in grams.

V. ACID COPPER SOLUTION

A. Copper Sulfate:

1. Pipette a 5 cc. sample into a 250 cc. erlenmeyer flask.
2. Add 100 cc. of distilled water and continue from II-C-4 on page 165.

The **COPPER SULFATE** ($CuSO_4 \cdot 5H_2O$) in oz./gal. is equal to:

$$\left\{ \frac{20.9}{\text{cc. of thiosulfate solution required for 0.20 g. Cu}} \right\} \times \text{cc. of thiosulfate required for sample of solution.}$$

The figures inside the brackets represent the factor for the standard thiosulfate solution. The copper sulfate can also be determined by taking the Baume reading. This gives the combined copper sulfate plus sulfuric acid from a table. Subtraction of the ounces of acid leaves the ounces of copper sulfate.

Baume Gravity of Solutions of Copper Sulfate Plus Sulfuric Acid

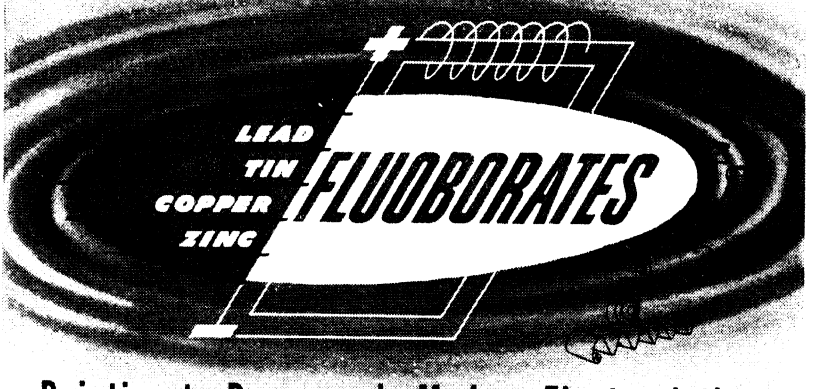
Baume	Copper Sulfate + Sulfuric Acid	Baume	Copper Sulfate + Sulfuric Acid
1.5	2.8 oz./gal.	14.5	24.7 oz./gal.
2.0	3.5 "	15.0	25.7 "
2.5	4.3 "	15.5	26.8 "
3.0	5.1 "	16.0	27.8 "
3.5	5.9 "	16.5	28.8 "
4.0	6.7 "	17.0	29.8 "
4.5	7.4 "	17.5	30.8 "
5.0	8.2 "	18.0	31.8 "
5.5	9.0 "	18.5	32.8 "
6.0	9.8 "	19.0	33.8 "
6.5	10.6 "	19.5	34.9 "
7.0	11.5 "	20.0	35.9 "
7.5	12.3 "	20.5	37.0 "
8.0	13.1 "	21.0	38.1 "
8.5	13.9 "	21.5	39.2 "
9.0	14.8 "	22.0	40.4 "
9.5	15.7 "	22.5	41.6 "
10.0	16.6 "	23.0	42.8 "
10.5	17.5 "	23.5	43.9 "
11.0	18.3 "	24.0	45.0 "
11.5	19.2 "	24.5	46.1 "
12.0	20.0 "	25.0	47.3 "
12.5	21.0 "	25.5	48.5 "
13.0	21.9 "	26.0	49.7 "
13.5	22.9 "	26.5	51.0 "
14.0	23.8 "	27.0	52.3 "

This method of obtaining the concentration of copper sulfate plus sulfuric acid is not accurate if other ingredients such as aluminum sulfate are present or if the solution is contaminated with iron.

B. Sulfuric Acid:

1. Pipette a 10 cc. sample into a 250 cc. erlenmeyer flask.
2. Add 150 cc. of distilled water and 5 drops of 0.2% methyl orange solution.
3. Titrate with standard 1.0 N sodium hydroxide (NaOH) solution until the color of the solution changes from violet to pale green. The 1.0 N NaOH

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is prepared by dissolving 40 g. of NaOH in distilled water and diluting to 1 liter. Exactly 2.00 g. of C.P. oxalic acid ($H_2C_2O_4 \cdot 2H_2O$) are weighed out and dissolved in 100 cc. of distilled water. A few drops of 2% phenolphthalein solution in alcohol are added and the solution is titrated with the NaOH until the solution changes from colorless to pink. 1 cc. of 1.0 N NaOH is equivalent to 0.0630 g. oxalic acid. Therefore the **normality of the NaOH** is equal to: 2/(cc. NaOH required for 2.00 g. oxalic acid $\times$ 0.0630) equals 31.7/(cc. of NaOH required for 2.00 g. oxalic acid). 1 cc. of 1.0 N NaOH is also equivalent to 0.0491 g. of H_2SO_4 so that **SULPHURIC ACID** (H_2SO_4) in oz./gal. is equal to:

$$\left\{ \frac{20.8}{\text{cc. NaOH required for 2.00 g. oxalic acid}} \right\} \times \text{cc. NaOH required for sample of solution}$$

The figures inside the brackets represent the factor for the standard sodium hydroxide solution.

VI. CYANIDE COPPER SOLUTION

A. Metallic Copper:

1. Proceed as in II-C on page 165.

B. Free Cyanide:

1. Proceed as in I-A on page 163.

C. Carbonates:

1. Proceed exactly as in III-C on page 171.

D. Rochelle Salt:

1. Proceed exactly as in II-E on page 167.

VII. CYANIDE GOLD SOLUTION

A. Metallic Gold:

1. Pipette a 10 cc. sample into a 250 cc. beaker.
2. Add 15 cc. of concentrated hydrochloric acid (HCl) under a hood and evaporate to a syrup over a water bath.
3. Add 150 cc. of distilled water.
4. Add 25 cc. of 20% potassium iodide (KI) solution and 2 cc. of 1% starch solution prepared as in II-C-8 on page 165.
5. Titrate with standard 0.01 N sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$) solution until the color disappears. The excess of thiosulfate is then titrated with standard 0.01 N iodine solution until a blue color appears.

The 0.01 N Sodium thiosulfate solution is prepared by dissolving 2.5 g. of the salt in 1 liter of distilled water. Exactly 0.05 g. of metallic gold (Au) are weighed out, dissolved in aqua regia and carefully evaporated to a syrup over a water bath. 150 cc. of distilled water, 25 cc. of 20% KI solution and 2 cc. of starch indicator are added and the solution is titrated with the thiosulfate until the color disappears. The excess thiosulfate is titrated with the iodine solution which is prepared by dissolving 2.5 g. of KI in 10 cc. of distilled water, adding 1.27 g. of iodine (I_2) and

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when this is dissolved, diluting to 1 liter. Pipette 10 cc. of the iodine solution into a 250 cc. erlenmeyer flask. Add 100 cc. of distilled water and 2 cc. of starch indicator. Titrate with the thiosulfate until the color disappears. Each cc. of the iodine solution is equivalent to 1/10 the number of cc. of thiosulfate used.

The exact number of cc. of thiosulfate required for the titration of 0.05 g. of gold will be equal to the total number of cc. used minus the excess, which is equal to the cc. of iodine solution used × the ratio between the iodine solution and the thiosulfate solution.

The **METALLIC GOLD** (Au) in troy oz./gal. is equal to:

$$\left\{\frac{0.608}{\text{cc. thiosulfate required for 0.05 g. Au}}\right\} \times \text{cc. thiosulfate required for sample.}$$

The figures inside the brackets represent the factor for the standard thiosulfate solution.

B. Free Cyanide:

1. Proceed as in I-A on page 163.

C. Carbonates:

1. Proceed exactly as in III-C on page 171.

VIII. IRON SOLUTION

A. Metallic Iron:

1. Pipette a 5 cc. sample into a 250 cc. erlenmeyer flask.
2. Add 125 cc. of distilled water and 15 cc. of sulfuric-phosphoric mixture which is made by diluting a mixture of 250 cc. sulfuric acid (H_2SO_4) and 100 cc. of phosphoric acid (H_3PO_4) (sp. gr.-1.7) to 1 liter.
3. Add a few drops of a solution of 1 g. diphenylamine in 100 cc. of 60% acetic acid ($HC_2H_3O_2$).
4. Titrate with standard 0.5 N potassium dichromate ($K_2Cr_2O_7$) until the color of the solution changes from green to an intense violet blue. The $K_2Cr_2O_7$ is prepared by weighing out exactly 24.52 g. of the dried salt, dissolving in distilled water and diluting to exactly 1 liter.

 1 cc. of 0.5 N $K_2Cr_2O_7$ is equivalent to 0.0279 g. of metallic iron (Fe).

The **METALLIC IRON** (Fe) in oz./gal. is equal to 0.744 × cc. of $K_2Cr_2O_7$ required for the sample.

B. Free Hydrochloric Acid:

1. Pipette a 25 cc. sample into a 250 cc. erlenmeyer flask.
2. Add 50 cc. of distilled water and 1 cc. of 0.1% bromphenol blue solution.
3. Titrate with 1.0 N NaOH prepared as in V-B-3 on page 176, until the solution changes to a blue color.

The **FREE HYDROCHLORIC ACID** (HCl) in oz./gal. is equal to:

$$\left\{\frac{6.16}{\text{cc. NaOH required for 2.00 g. oxalic acid}}\right\} \times \text{cc. of NaOH required for sample of solution.}$$

The figures inside the brackets represent the factor for the standard sodium hydroxide solution.

CHROMIC ACID

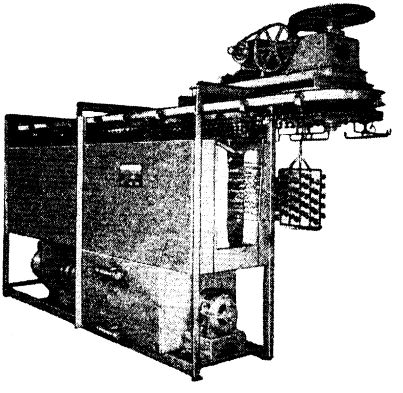
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IX. LEAD SOLUTION

A. Metallic Lead:

1. Pipette a 2 cc. sample into a 250 cc. beaker.
2. Add 100 cc. of distilled water and heat almost to boiling.
3. Add 10 cc. of 20% sulfuric acid (H_2SO_4) solution, slowly while stirring. Let stand until cool.
4. Filter, using a weighed Gooch crucible with an asbestos mat. Wash.
5. Dry and ignite the crucible. Cool in a dessicator and weigh.

The **METALLIC LEAD** (Pb) in oz./gal. is equal to:

45.5 × gain in weight of the crucible in grams.

X. WHITE NICKEL SOLUTION

A. Metallic Nickel:

1. Pipette a 5 cc. sample into a 250 cc. erlenmeyer flask.
2. Add 75 cc. of a solution made up by dissolving 50 g. of sodium pyrophosphate ($Na_4P_2O_7 \cdot 10H_2O$), 50 g. of ammonium chloride (NH_4Cl) and 2 g. of potassium iodide (KI) in water and diluting to 1 liter.
3. Add concentrated ammonium hydroxide (NH_4OH) drop by drop until the solution shows a bluish color and smells faintly of ammonia. About 3 drops are necessary for a high pH solution.
4. Titrate with standard 0.5 N sodium cyanide (NaCN) solution until the precipitate which first appears just redissolves.

The standard NaCN is prepared by dissolving 25 g. of NaCN and 1.7 g. of silver nitrate ($AgNO_3$) in 1 liter of distilled water. Exactly 0.20 g. of pure metallic nickel are weighed out and dissolved in a 250 cc. erlenmeyer flask with a mixture of 5 cc. of concentrated sulfuric acid, 5 cc. of concentrated nitric acid, and 5 cc. of distilled water. The solution is evaporated until dense white fumes of sulfur trioxide (SO_3) are evolved. Cool, add a slight excess of NH_4OH and boil. When the solution smells faintly of ammonia, cool to room temperature, add 75 cc. of the chloride-pyrophosphate mixture and titrate with the standard NaCN until the precipitate formed redissolves.

The **METALLIC NICKEL** (Ni) in oz./gal. is equal to:

$$\left\{ \frac{5.34}{\text{cc. of NaCN solution required for 0.20 g. Ni}} \right\} \times \text{cc. NaCN required for sample of solution.}$$

The figures inside the brackets represent the factor for the standard sodium cyanide solution.

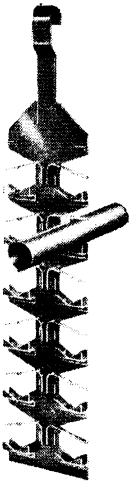
B. Chlorides:

1. Pipette a 5 cc. sample into a 250 cc. erlenmeyer flask.
2. Add 50 cc. of distilled water and 1 cc. of 2% sodium chromate (Na_2CrO_4) solution.
3. Titrate with standard 0.1 N silver nitrate ($AgNO_3$) solution prepared as I-A-3 on page 163, until a red precipitate forms.

1 cc. of 0.1 N $AgNO_3$ is equivalent to:
0.00535 g. ammonium chloride (NH_4Cl)
0.00585 g. sodium chloride (NaCl)
0.0119 g. nickel chloride ($NiCl_2 \cdot 6H_2O$)

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The **chloride expressed as nickel chloride** in oz./gal. is equal to:

0.317 × cc. of $AgNO_3$ required for sample.

Note: If the pH of the solution is below about 4.5, add 50 cc. of distilled water and 8 drops of bromcresol purple indicator, then add dropwise 2% ammonia solution until the color changes from a yellow green to a bluish green.

C. Boric Acid:

1. Pipette a 5 cc. sample into a 250 cc. erlenmeyer flask.
2. Add 25 cc. of C.P. glycerine [$C_3H_5(OH)_3$] and ½ cc. of bromcresol purple indicator.
3. Titrate with standard 0.1 N solution hydroxide (NaOH) solution until the color changes to dark green and then to purple.

The standard 0.1 N NaOH is prepared as follows:

4 grams of NaOH are dissolved in distilled water and diluted to 1 liter. Exactly 0.20 g. of C.P. oxalic acid ($H_2C_2O_4 \cdot 2H_2O$) is weighed out and dissolved in 100 cc. of distilled water. A few drops of bromcresol purple are added and the solution is titrated with the NaOH solution until the solution turns blue.

One cc. of 1.0 N NaOH is equivalent to 0.0619 g. of boric acid so that the **BORIC ACID** (H_3BO_3) in oz./gal. is equal to:

$$\left\{ \frac{5.25}{\text{cc. of NaOH required for 0.20 g. oxalic acid}} \right\} \times \text{cc. of NaOH required for sample.}$$

The figures inside the brackets represent the factor for the standard sodium hydroxide solution.

XI. BLACK NICKEL SOLUTION

A. Metallic Nickel:

1. Proceed exactly as in X-A on page 183.

B. Metallic Zinc:

1. Pipette a 10 cc. sample into a 250 cc. beaker.
2. Add 3% sodium cyanide (NaCN) solution until the precipitate which first forms dissolves completely and heat.
3. Continue from II-D-3 on page 167.

C. Sodium Thiocyanate:

1. Pipette a 10 cc. sample into a 250 cc. erlenmeyer flask.
2. Add 100 cc. of distilled water.

PLATING and JOB MACHINE WORK

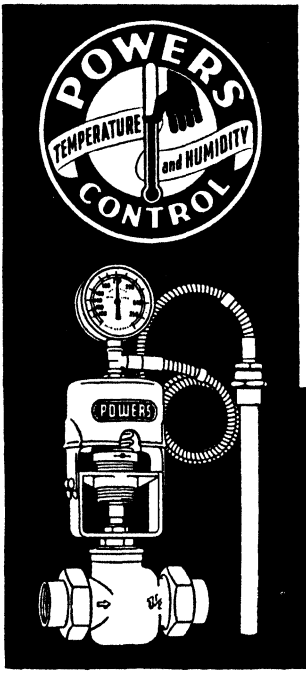
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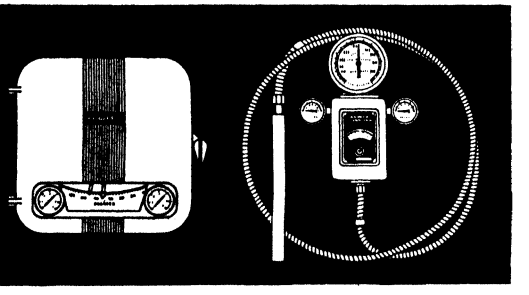
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3. Add sulfuric acid (H_2SO_4) drop by drop until the solution turns blue litmus red.
4. Add 15 cc. of a 2% ferric ammonium sulfate [$Fe_2(SO)_4)_3 \cdot (NH_4)_2SO_4 \cdot 24H_2O$] solution and titrate with standard 0.1 N silver nitrate ($AgNO_3$) prepared as in I-A-3 on page 163, until the red color disappears. 1 cc. of 0.1 N $AgNO_3$ is equivalent to 0.00811 g. of sodium thiocyanate (NaCNS).

The **SODIUM THIOCYANATE** (NaCNS) in oz./gal. is equal to: 0.108 × cc. of $AgNO_3$ required for sample of solution.

XII. SILVER SOLUTION

A. Metallic Silver

1. Pipette a 10 cc. sample into a 250 cc. erlenmeyer flask.
2. Add 20 cc. of concentrated sulfuric acid (H_2SO_4) and 5 cc. of nitric acid (HNO_3) **while shaking under a hood.** (This operation must be carried out under a hood, because poisonous cyanide fumes are evolved.)
3. Boil until all the white precipitate has dissolved.
4. Cool to room temperature.
5. Add 100 cc. of distilled water and allow to cool to room temperature.
6. Add 1 cc. of 2% ferric ammonium sulfate [$Fe_2(SO_4)_3 \cdot (NH_4)_2SO_4 \cdot 24H_2O$] solution and titrate with standard 0.1 N sodium thiocyanate (NaCNS) solution until a faint pink color appears.

The standard NaCNS is prepared by dissolving 10 g. of the salt and 0.5 cc. of 48% formaldehyde in 1 liter of distilled water. Pipette 10 cc. of standard 0.1 N $AgNO_3$ solution, prepared as in I-A-3 on page 163 into a 250 cc. erlenmeyer flask. Add 100 cc. of distilled water and 1 cc. of 2% ferric ammonium sulfate solution and titrate with the NaCNS until a faint pink color appears.

10 cc. of $AgNO_3$ solution contain 0.1079 g. of metallic silver (Ag).

The **METALLIC SILVER** (Ag) in troy oz./gal. in the plating solution is equal to:

$$\left\{ \frac{1.31}{\text{cc. NaCNS required for 10 cc. } AgNO_3} \right\} \times \text{cc. NaCNS required for sample of solution.}$$

The figures inside the brackets represent the factor for the standard thiocyanate solution.

B. Free Cyanide:

1. Pipette a 10 cc. sample into a 250 cc. erlenmeyer flask.
2. Add 100 cc. of distilled water and 1 cc. of 10% potassium iodide (KI) solution.
3. Titrate with standard 0.1 N $AgNO_3$ solution, prepared as in I-A-3 on page 163 until a faint yellowish turbidity is produced.

The **FREE SODIUM CYANIDE** (NaCN) in oz./gal. is equal to: 0.131 × cc. of $AgNO_3$ required for sample of solution.

C. Carbonates:

1. Proceed exactly as in III-C on page 171.

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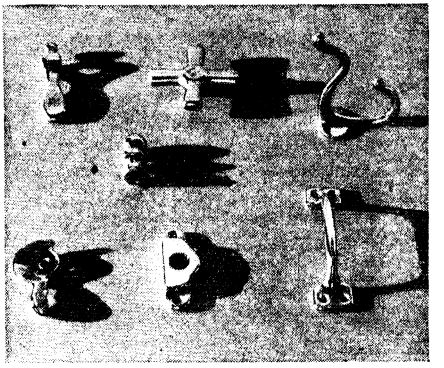
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XIII. TIN SOLUTION

A. Sodium Stannate:

1. Pipette a 5 cc. sample into a 500 cc. erlenmeyer flask.
2. Add 70 cc. of concentrated hydrochloric acid (HCl), 200 cc. of distilled water and about ⅛ teaspoonful of C.P. iron powder.
3. Boil until all the iron has gone into solution.
4. Close the flask with a stopper and cool to room temperature under cold running water.
5. Add 2 cc. of starch indicator, prepared as in II-C-8 on page 165 and titrate with standard 0.25 N iodine solution until a permanent color appears.

The standard iodine solution is prepared as follows:

60 g. of potassium iodide (KI) are dissolved in 200 cc. of distilled water. 32 g. of pure iodine (I_2) are added and the whole diluted to 1 liter after the iodine has dissolved. Exactly 0.25 g. of C.P. tin (Sn) is weighed out and dissolved in 70 cc. of concentrated HCl by boiling in a 500 cc. erlenmeyer flask. As soon as the tin is dissolved, 200 cc. of distilled water and about ⅛ teaspoonful of iron powder are added. The solution is then treated as above, proceeding from XII-A-3.

The **METALLIC TIN** (Sn) in oz./gal. equals:

$$\left\{\frac{6.68}{\text{cc. iodine required for 0.25 g. of tin}}\right\} \times \text{cc. of iodine required for sample of solution.}$$

The figures inside the brackets represent the factor for the standard iodine solution.

B. Free Sodium Hydroxide:

1. Pipette a 10 cc. sample into a 250 cc. erlenmeyer flask.
2. Add 10 cc. of distilled water and 8 drops of LaMotte Sulfo-Orange Indicator.
3. Titrate with standard 1.0 N hydrochloric acid (HCl) solution prepared as in III-C-7 on page 171 until the color changes from orange to yellow.

The **FREE SODIUM HYDROXIDE** (NaOH) in oz./gal. equals:

[0.535 × Normality of HCl] × cc. of HCl required for sample of solution.

The figures inside the brackets represent the factor for the standard hydrochloric acid solution.

XIV. ACID ZINC SOLUTION

A. Metallic Zinc:

1. Pipette a 2 cc. sample into a 250 cc. beaker.
2. Add 12 cc. of concentrated hydrochloric acid (HCl) and boil for 1 minute.
3. Add 115 cc. of distilled water and 25 cc. of 20% ammonium chloride (NH_4Cl) solution. Heat almost to boiling.
4. Titrate with the standard ferrocyanide solution as in II-D-6 on page 167.

The **METALLIC ZINC** (Zn) in oz./gal. is equal to:

$$\left\{\frac{16.7}{\text{cc. of ferrocyanide soln. required for 0.25 g. zinc}}\right\} \times \text{cc. of ferrocyanide solution required for sample of solution.}$$

The figures inside the brackets represent the factor for the standard ferrocyanide solution.

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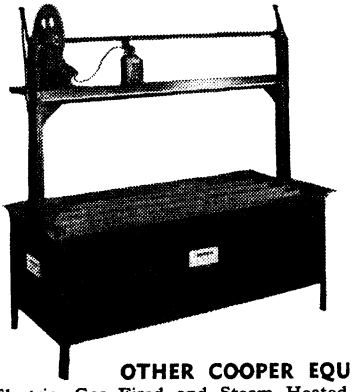
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B. Acidity:

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XV. CYANIDE ZINC SOLUTION

A. Metallic Zinc:

1. Pipette a 5 cc. sample into a 250 cc. beaker.
2. Add 12 cc. of concentrated hydrochloric acid (HCl) **while shaking under a hood.** (This operation must be carried out under a hood because poisonous cyanide fumes are evolved). Add 20 cc. of 25% of ammonium chloride (NH_4Cl) and 120 cc. of distilled water, then heat almost to boiling.
3. Titrate with the standard ferrocyanide solution as in II-D-6 on page 167.

The **METALLIC ZINC** (Zn) in oz./gal. is equal to:

$$\left\{\frac{6.68}{\text{cc. ferrocyanide soln. required for 0.25 g. zinc}}\right\} \times \text{cc. of ferrocyanide solution required for sample of solution.}$$

The figures inside the brackets represent the factor for the standard ferrocyanide solution.

B. Total Cyanide:

1. Pipette a 5 cc. sample into a 250 cc. erlenmeyer flask.
2. Add 100 cc. of distilled water, 5 cc. of 20% sodium hydroxide (NaOH) solution and 5 cc. of 10% potassium iodide (KI) solution.
3. Titrate with the standard silver nitrate solution prepared as in I-A-3 on page 163 until a faint yellow turbidity is produced.

The **TOTAL SODIUM CYANIDE** (NaCN) in oz./gal. is equal to:

0.261 × cc. $AgNO_3$ required for sample of solution.

C. Sodium Hydroxide:

1. Pipette a 10 cc. sample into a 250 cc. erlenmeyer flask.
2. Add 10 cc. of 10% sodium cyanide (NaCN) solution and 8 drops of LaMotte Sulfo-Orange Indicator.
3. Titrate with standard 1.0 N hydrochloric acid (HCl) solution prepared as in III-C-7 on page 171 until the color changes from orange to yellow.

The **SODIUM HYDROXIDE** (NaOH) in oz./gal. equals:

[0.535 × Normality of HCl] × cc. of HCl required for sample of solution.

The figures inside the brackets represent the factor for the standard hydrochloric acid solution.

D. Carbonates:

1. Proceed exactly as in III-C on page 171.



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SOLUTIONS FOR STRIPPING METALLIC COATINGS

By G. B. Hogaboom, Jr. and Nathaniel Hall

CADMIUM

Cadmium from steel, brass or copper:

1. Hydrochloric acid	1 gal.	Simple immersion.	
Antimony trioxide	2 oz.	Room temperature.	
Water	½ pint		

A smut of antimony is produced on the work.

2. Ammonium nitrate	1 lb.	Immersion at room temp.
Water to make	1 gal.	

COPPER

Copper from non-ferrous metals:

The following formula has been suggested by Bell Laboratories:

Sodium sulphide	28 oz.
Sulphur	2 "
Water to make	1 gal.

Boil to dissolve sulfur. Use at room temperature. Immerse work for 5 minutes. Brush off loose sulfide. Repeat several times. Rinse and dip in 10% sodium cyanide solution. Brush. If any deposit remains, repeat whole procedure.

Copper from zinc:

1. Sodium sulfide	1 lb.	Make work anode, 2 volts.
Water to make	1 gal.	

2. Stoddard Method:

Chromic acid	200-259 g./l.
Ratio chromic acid to sulfate (sulfuric acid)	100:1
Temperature	20-25° C.
Current density	7-14 amp./dm.^{2} (6 to 12 volts)
Alternating current	50 to 60 cycles

Note: Any sulfide such as polysulfide, oxidizing liquid, liquid sulfur, liver of sulfur, etc., may be used for removal of copper by forming copper sulfide.

CHROMIUM

Chromium from steel or nickel:

Sodium hydroxide	6 oz.	Reverse current at 6 volts.
Water to make	1 gal.	Room temperature.

Chromium from brass, copper, or nickel:

1. Hydrochloric acid	1 pint	Temp. 125° F.
Water to make	1 gal.	
2. Hydrochloric acid, conc.		Room temperature.

3. Sulfuric acid strip, (as for nickel from steel), but nickel will also be removed.

CYANIDE SOLUBLE METALS

Brass, bronze, cadmium, copper, zinc, gold and silver deposits from steel:

Sodium cyanide	12 oz.	Room temperature.	
Sodium hydroxide	2 "	Voltage	6 volts (work anode)
Water to make	1 gal.	Steel cathodes.	

GOLD

There is no good method for removing gold without causing some attack on the underlying metal. The following solution can be used for removing light deposits on copper and its alloys and heavy deposits from high nickel alloys and ferrous metals.

1. Sodium cyanide 2 oz. Water 1 pt.

To one pint of this solution, add ½ fluid ounce of 100 vol. hydrogen peroxide. Then immerse the article to be stripped. The container should be placed in a sink as there may be a sudden evolution of heat with violent gassing. This will increase the stripping action. After gassing ceases, add more peroxide cautiously. If solution turns blue (from copper) or is not clear, more cyanide is needed.

Only a few pieces at a time should be stripped otherwise the rapid action on a large amount of work in a large volume of solution will cause boiling over.

Gold from copper, copper alloys, and zinc:

2. U. S. Pat. 2,185,858. S. R. Mason to Western Electric Co.

Make work anode in conc. sulfuric acid. Temp. must be below 100°F. May add small amount chromic acid or single nickel salts to help the action.

NICKEL

Nickel from steel:

1. Sulfuric acid 5 pts. Room temperature.
 Glycerine 1 oz. Reverse current, 6 volts.
 Water 3 pts. Lead cathode.

The rate of stripping will be increased by adding more water, but this will increase the tendency to pit. Copper sulphate (crystals), 4 oz./gal. may be added instead of glycerine to reduce pitting of steel.

2. Fuming nitric acid. Simple immersion at room temperature.

Put work in dry. Keep covered when not in use to keep out water.

or: Concentrated nitric acid 1 gal. Hydrochloric acid 0.1 fluid oz.

(U. S. Pat. 2,200,486. E. F. Burdick to Western Electric Co.)

3. Sodium nitrate 4-5 lb./gal. Current density 100 amp./ft.^{2} minimum. Reverse current at 200°F. and 6 volts.

Use steel cathodes and keep pH between 6 and 8.

Work should be agitated in any stripping operation to prevent pitting.

Nickel from brass or copper:

1. Sulfuric acid strip, as for nickel from steel.
2. Hydrochloric acid 6 oz. Room temperature.
 Water 1 gal. Reverse current.
 Gas carbon cathodes.
3. Light deposits of nickel may be removed in a sulfuric-nitric bright dip.

SILVER

Silver, from brass, or nickel silver:

Sulfuric acid 19 parts by vol. Simple immersion at 180°F. Keep
Nitric acid 1 part by vol. water out.

Silver, from white metal:

Sodium cyanide 4 oz. Reverse current, 4 volts.
Water to make 1 gal.

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Engineers at General Electric looked over the field and chose Luster-on* for the protective bright dip for zinc plated parts on their new 1946 radios. Read their statement — it tells the whole story of Luster-on superiority. Then send the coupon and a sample part for proof that you can see with your own eyes.



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Please send me full information and free sample of STRIPODE for better nickel stripping.

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Address

Tin from steel, copper, or brass:

Use hydrochloric acid, and antimony oxide, as under cadmium strip from steel.

Tin from brass, bronze and copper:

1. Ferric chloride 10-14 oz./gal.
 Copper sulfate 18-21 "
 56% Acetic acid 40-60 fl. oz./gal.

 The solution is reactivated by additions of hydrogen peroxide to oxidize the reduced iron.

2. Sodium hydroxide 16 oz./gal.

 Room temp. Reverse current at 6 volts.

HEATING OF SOLUTIONS IN THE PLATING ROOM

By G. B. Hogaboom, Jr. and Nathaniel Hall

CALCULATIONS

To find the amount of electricity, gas, or steam needed first find the B.t.u. requirement of each tank. (B.t.u. = British thermal unit = Amount of heat which will raise the temperature of 1 lb. of water 1°F.)

For practical purposes figure all solutions as plain water. A gallon of water weighs 8.33 lbs. Multiplying pounds of water in the tank by desired temperature rise gives B.t.u. requirement. Example:

Cleaner tank 36" x 24" x 30".

$$\textit{Capacity} = \frac{36 \times 24 \times 30}{231} = 112 \text{ gals.}$$

Weight of solution = 112 × 8.33 = 934 lbs.

Temperature rise desired = From say 60°F. to 210°F. = 150°F.

B.t.u. requirement = 934 × 150 = 140,000 B.t.u., plus 10% to allow for heat lost from surface of solution (which is major loss), and sides of tank during heating up period = 154,000 B.t.u.

B.t.u. delivered by different methods are:

Steam boiler: 1 Boiler horsepower = 33,000 B.t.u. per hour.

City gas, also called carburetted water gas: Generally about 530 B.t.u. per cubic foot.

Electricity: 1 Kilowatt hour = 3412 B.t.u.

In example given above, 154,000 B.t.u. are needed, and if it is desired to heat up in 1 hour the heat source must deliver 154,000 B.t.u. per hour, which would be as follows, for the different methods:

$$\textit{Steam boiler:} \frac{154{,}000}{33{,}000} = 4.7 \text{ Boiler Horsepower.}$$

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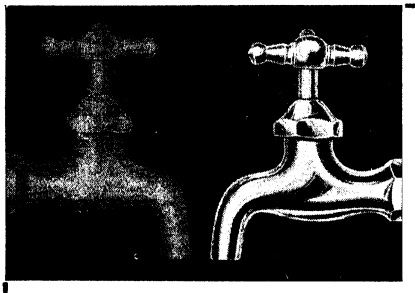
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Gas: $\frac{154{,}000}{530} = 290$ cubic feet per hour. To accomplish this will require 3 - 2½" pipe size immersion burners. See table below.

Electricity: $\frac{154{,}000}{3412} = 45$ kilowatts.

The size and cost of a heating plant to heat up all solutions in a plating room at the same time in 1 hour may prove considerable. Progressive heating of the tanks can sometimes be worked out. The size can be cut in half by allowing a 2 hour heating up period, and in quarter for a 4 hour period. Extending the time beyond 4 hours should be considered with caution as the capacity of the heating unit may then be insufficient to keep up with losses from tank surfaces plus the heat taken up by work passing through the tank.

THE APPLICATION OF DIFFERENT HEATING METHODS

STEAM: FEET OF STEEL PIPE NECESSARY FOR TANK COIL TO HEAT 1 GAL. OF WATER FROM 60 TO 210° F. IN 1 HOUR[1]

	Steam pressure. Gauge reading, lbs.					
Pipe Size	**5**	**10**	**25**	**50**	**100**	**150**
½"	.56	.41	.30	.23	.17	.14
¾"	.42	.31	.23	.17	.13	.10
1"	.35	.25	.18	.13	.10	.08
1¼"	.25	.19	.14	.10	.075	.061
1½"	.22	.16	.12	.087	.064	.052
2"	.17	.13	.09	.068	.050	.041
2½"	.14	.11	.076	.057	.042	.034

For lead pipe, add half again as much footage.

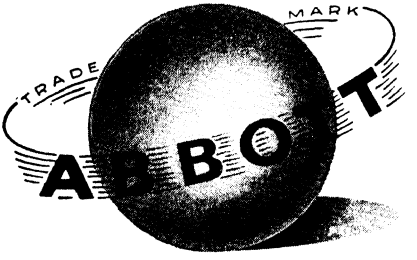
GAS: REPRESENTATIVE CAPACITIES OF BURNERS:[2]

Drilled pipe burners			Immersion burners	
Pipe size	**Flame length**	**Cu. ft. gas per hour**	**Pipe size of Coil**	**Cu. ft. of gas per hour**
½"	12"	16	1¼"	18
¾"	12"	21	1½"	30
¾"	24"	35	2"	50
1"	12"	30	2½"	100
1"	24"	41	3"	125
1"	48"	73	4"	200
1½"	36"	105		
2"	48"	190		

ELECTRICITY:

The kilowatt capacity needed for a tank is best applied not by one but by two or more units which add up to the desired total. This allows turning off all units when temperature has been reached except one or two needed to maintain temperature. Controllers placed on the one or two temperature holding units need not be as large as would be required if a controller were installed for the total capacity.

1. R. W. Mitchell, Metal Cleaning Handbook.
2. Charles A. Hones, Inc., Baldwin, N. Y.



BURNISHING

BALLS — BARRELS — MATERIALS

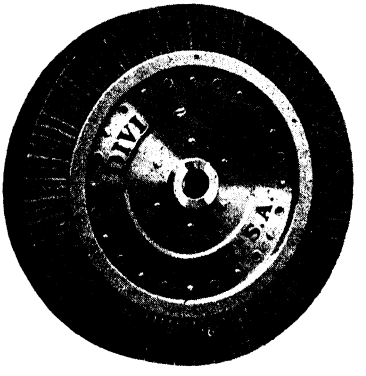
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PLATING TANK WIRING

By G. B. Hogaboom, Jr. and Nathaniel Hall

Newark, N. J.

Fig. 1 represents a typical wiring diagram for a plating tank, including rheostat, meters and switches.

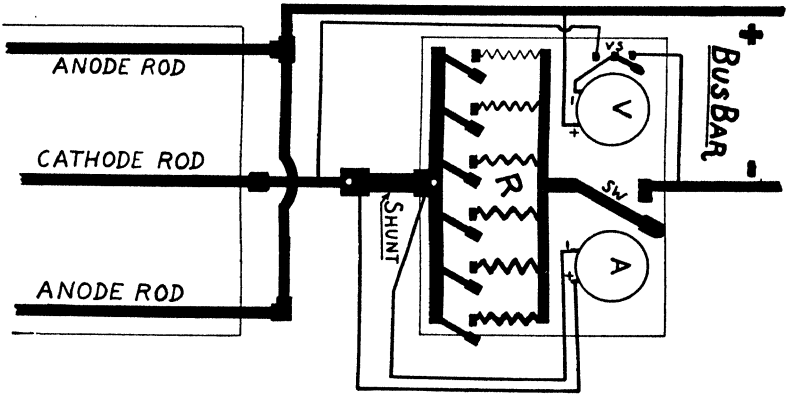


Figure 1

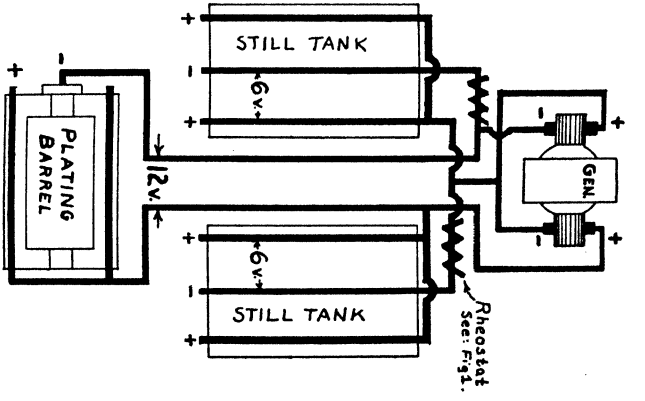


Figure 2. Three-wire system enabling the use of both 6 and 12 volts.

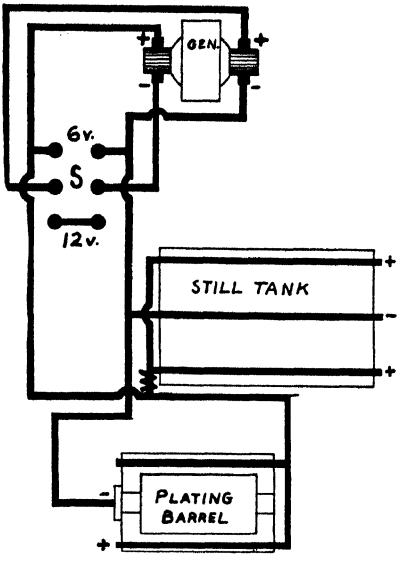


Figure 3 (left). Diagram illustrating how 6 and 12 volts may be obtained using a double pole, double throw switch.

The switch SW is known as the master switch and is used for breaking the tank circuit without affecting the position of the rheostat switches. Without this switch, it is necessary to open all the rheostat switches when unloading the tank. Although not shown in the illustration, this switch is sometimes mounted as a double throw switch, the third terminal being connected to the opposite end of the rheostat so that the current, instead of passing through the resistance coils, is passed directly to the tank, thus short circuiting the resistance coils. This arrangement is used where the full line voltage is desired for striking.

The small single pole double throw switch VS is used so that one voltmeter can be employed for both bus bar voltage with the switch up and tank voltage with the switch down. When there is no work in the tank, the two readings should be identical since the minute amount of current passing through the resistance coils to activate the meter does not result in enough of a voltage drop to register on the instrument.

The ammeter A is supplied with a pair of calibrated leads and these should be used with the accompanying shunt. The shunt acts as a bypass so that all the current will not be required to pass through the ammeter which obviously would not be practical in the case of large amounts of current, requiring large conductors in the coil windings.

It will be noted that the rheostat is connected in the negative line. This is the usual practice since there is then no doubt that the current indicated on the ammeter is the current flowing through the work. There is an advantage in connecting it in the positive line, however, in that the tank is completely disconnected when no work is in the tank and the switches are open. With the rheostat in the negative line, the positive bus bar goes directly to the anode rod and current is conducted into the anodes. If there is a ground on the tank, the current may pass from the anode to the tank wall and into the ground, with the result that the sides

of the tank may plate up with metal. This, of course, is of importance only when the tank is of metal and unlined. The advantages of both hookups may be obtained by placing the rheostat in the positive line and the ammeter shunt in the negative line.

Rheostats are ordered according to voltage drop and ampere capacity. The voltage drop is the difference between the line voltage and the plating voltage. For instance, if the line voltage is 6 volts, it is necessary to order a 4 volt drop rheostat for nickel plating at 2 volts.

When a double commutator generator is available, it is possible to use both 6 and 12 volts by means of either a three wire system as shown in Fig. 2, or by the use of a double pole double throw switch as in Fig. 3. In Figure 2, it will be noted that the positive of one commutator is coupled with the negative of the other commutator and a bus bar is drawn from this connection, called the neutral bus. The voltages available are 6 volts between the neutral bus and either the positive or negative bus bar, and 12 volts between the positive and negative. It is thus possible to operate a barrel at 12 volts and still tanks at 6 volts, *at the same time*, from the same generator.

It will be noted that each of the still tanks in Fig. 2 are fed from different commutators. This is the important disadvantage of the three wire system since very often one tank draws more current than the other and the unbalancing of the commutators results in severe sparking. With this system, since the two commutators are connected permanently in series, it is not possible to draw the full amperage of the generator into one still tank at 6 volts; in fact it is not possible to draw more than ½ of the amperage without overloading the commutator.

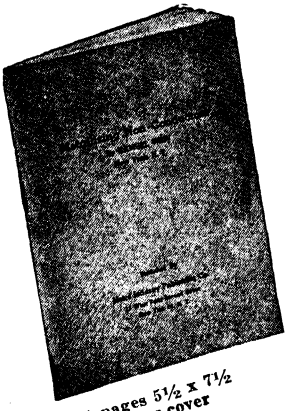
The three wire system is rarely used in up-to-date plants because of these disadvantages, it being the usual practice to provide separate generators for the still tank plating and for the barrels.

If the plating barrels and the still tanks are not used at the same time, which is the common situation in small plants, the arrangement in Fig. 3 is most satisfactory since by throwing the switch "S" up, the full capacity of the generator is available in any tank at 6 volts and by throwing the switch down, the commutators may be placed in series to deliver 12 volts for barrel operation. The capacity of the switch need be only half the amperage capacity of the generator at 6 volts. Thus, if the generator is a 1,000 ampere/6 volt machine, the capacity of the switch need be only 500 amperes.



Metallizing Non-Conductors

By SAMUEL WEIN



64 pages 5½ x 7½
paper cover

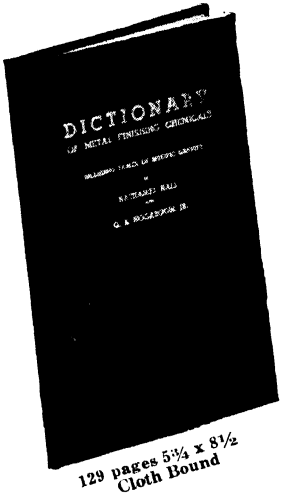
The present work deals with every known method for "metallizing" or the deposition of metals by electrolysis (plating) on non-conductors. It is divided into several sections, i. e., those processes which use chemical, mechanical and physical methods for treatment of surfaces for metallizing. In these groups the specific methods are chronologically reviewed and so the reader can very readily get a better idea of the progress made by the various workers in these arts. At the end is an alphabetical listing of contributors to the art, so that the serious workers can refer to the original sources of the information given in the text by Mr. Wein.

The text is prepared in a practical fashion so that the formulas given will be of material use and is the result of literature collected by the author for more than 25 years and which has been in use by a number of industrial concerns here in the United States and abroad.

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By NATHANIEL HALL and G. B. HOGABOOM, JR.



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TABLES

AVERAGE CATHODE CURRENT EFFICIENCIES OF COMMON PLATING SOLUTIONS

By Nathan E. Promisel

Washington, D. C.

Note: Some of these figures are very approximate. Cathode efficiency depends very much on the plating conditions actually used. However, the figures below serve as a starting point for estimating metal yields from tables of plating times or electrochemical equivalents (see page 207) which are based on cathode efficiencies of 100%.

Metal	Type of Bath	Usual Cathode Efficiency—per Cent
Cadmium	Cyanide	88- 95
Chromium	Chromic acid-Sulfate	12- 16
Copper	Acid Sulfate	97-100
Copper	Cyanide	30- 60
Copper	Rochelle-Cyanide	40- 70
Cobalt	Acid Sulfate	95- 98
Gold	Cyanide	70- 90
Indium	Cyanide	30- 50
Indium	Fluoborate	30- 50
Indium	Acid Sulfate	70- 80
Iron	Acid Chloride	90- 98
Iron	Acid Sulfate	95- 98
Lead	Fluoborate	100
Lead	Fluosilicate	100
Nickel	Acid Sulfate	94- 98
Silver	Cyanide	100
Tin	Acid Sulfate	90- 95
Tin	Stannate	70- 90
Rhodium	Acid Phosphate	10- 18
Rhodium	Acid Sulfate	10- 18
Zinc	Acid Sulfate	99
Zinc	Cyanide	85- 90

TEMPERATURE—STEAM PRESSURE TABLE

Temperature °F.	Absolute* Steam Pressure lb./in.^{2}	Temperature °F.	Absolute* Steam Pressure lb./in.^{2}
110	1.274	260	35.43
120	1.692	270	41.85
130	2.221	280	49.2
140	2.887	290	57.55
150	3.716	300	67.01
160	4.739	310	77.68
170	5.990	320	89.65
180	7.510	330	103.03
190	9.336	340	117.99
200	11.525	350	134.62
210	14.123	360	153.01
220	17.188	370	173.33
230	20.78	380	195.70
240	24.97	390	220.29
250	29.82	400	247.25

*To obtain gauge pressure subtract 14.7 from the absolute pressure.

ELECTRICAL RESISTIVITY OF METALS AND ALLOYS

(Ohm-centimeters $\times$ 10^{-6})

Aluminum (Commercial)	2.83	Mercury	95.8
Brass	6.4-8.4	Monel	42
Bronze	13-18	Nichrome	100
Cadmium	7.5	Nickel	7.8
Carbon	3500	Nickel-silver (18%)	33
Chromium	2.6	Palladium	11
Cobalt	9.7	Phosphor bronze	5-10
Copper	1.75	Platinum	10
Gold	2.44	Rhodium	4.7
Gold (14 Kt.)	13.2	Silver	1.63
Graphite	800	Steel (Soft)	16
Indium	8.37	Steel (Hard)	45
Iron (Pure)	10	Steel (Stainless 18/8)	40-50
Lead	22	Tin	11.5
Magnesium	4.6	Zinc	5.8

ELECTROCHEMICAL EQUIVALENTS

Calculated on Basis of 100% Cathode Efficiency

Metal	Symbol	Valence	Atomic Weight	Specific Gravity	Thickness in Inches of 1 oz./sq. ft.	Grams Deposited Per Amp. Hr.	Amp. Hr. Per Sq. Ft. to Deposit 0.001"
Antimony	Sb	3	121.76	6.68	.00180	1.514	10.4
Cadmium	Cd	2	112.41	8.65	.00139	2.0968	9.73
Chromium	Cr	6	52.01	7.1	.00169	0.3233	51.8
Cobalt(ous)	Co	2	58.94	8.9	.00135	1.100	19.0
Copper(ous)	Cu	1	63.57	8.93	.00134	2.371	8.89
Copper(ic)	Cu	2	63.57	8.93	.00134	1.186	17.8
Gold(ous)	Au	1	197.2	19.3	.00068*	7.356	6.2
Gold(ic)	Au	3	197.2	19.3	.00068*	2.450	18.6
Hydrogen	H	1	1.0078	0.0899 g./l.	——	0.0376	—
Indium	In	3	114.76	7.31	.00182*	1.427	12.0
Iron(ous)	Fe	2	55.84	7.87	.00153	1.042	17.9
Lead	Pb	2	207.22	11.35	.00106	3.865	6.9
Nickel	Ni	2	58.69	8.90	.00135	1.095	19.0
Oxygen	O	2	16.00	1.429 g./l. (0°C.)	——	0.2985	—
Palladium	Pd	2	106.7	11.40	.00116*	1.990	13.5
Platinum	Pt	4	195.23	21.45	.00062*	1.821	27.8
Rhodium	Rh	3	102.91	12.5	.00106*	1.280	22.9
Silver	Ag	1	107.88	10.5	.00126*	4.025	6.2
Tin(ous)	Sn	2	118.70	7.3 (tetragonal)	.00164	2.214	7.8
Tin(ic)	Sn	4	118.70	7.3 "	.00164	1.107	15.6
Zinc	Zn	2	65.38	7.14	.00168	1.182	14.3

*These figures are for 1 troy ounce per square foot.

CONVERSION TABLES

All quantities refer to U. S. units

Carat (c) (metric)	3.08647 grains; 0.2 gram.
Centimeter (cm.)	0.3937 inch; 0.03281 ft.; 0.01 meter.
Cubic Centimeter ($cm.^3$)	0.03381 oz. (fluid); 0.061023 cubic inch; 0.001 liter.
Cubic Foot ($ft.^3$ or cu. ft.)	7.481 gallons; 1728 cubic inches; 23.316 liters; 28,317 cubic centimeters.
Cubic Inch ($in.^3$ or cu. in.)	0.004329 gallon; 0.5541 ounce (fluid); 16.3872 cubic centimeters.
Foot (ft.)	0.3048 meter; 30.48 centimeters.
Gallon (gal.)	weighs 8.337 lb. at 62°F.; 0.13368 cubic foot; 128 oz. (fluid); 231 cubic inches; 3.7853 liters; 3785.4 cubic centimeters.
Grain (gr.)	0.0020833 oz. (troy); 0.0022857 oz. (avoirdupois); 0.0648 gram.
Gram (g.)	0.03215 oz. (troy); 0.035274 oz. (avoirdupois); 0.643 pennyweight.
Inch (in.)	2.540 centimeters.
Kilogram (kg.)	2.2046 lb. (avoirdupois); 1000 grams.
Liter (l.)	0.2642 gal.; 0.035316 cu. ft.; 1.0567 quarts (liquid); 61.025 cu. in.; 1000 cubic centimeters.
Mil	0.001 inch; 0.00254 centimeter; 25.4 microns.
Milligram (mg.)	0.001 gram.
Millimeter (mm.)	0.03937 inch; 0.001 meter; 1000 microns.
Ounce (fluid) (fl. oz.)	0.0078125 gallon; 0.03125 quart (liquid); 1.80469 cubic inches; 29.5737 cubic centimeters.
Ounce (avoirdupois) (oz.)	0.0625 lb. (avoir.); 0.911458 oz. (troy); 18.2292 pennyweights; 437.5 grains; 28.3495 grams.
Ounce (troy)	0.068571 lb. (avoir.); 0.0833 lb. (troy); 20 pennyweights; 480 grains; 31.10348 grams.
Pennyweight (dwt.) (troy)	0.0034286 lb. (avoir.) 0.004167 lb. (troy); 1/20 oz. (troy); 24 grains; 1.552 grams.
Pint (pt.) (liquid)	16 oz. (fluid); 28.875 cu. in.; 473.18 cubic centimeters.
Pound (avoirdupois) (lb. av.)	1.2153 lb. (troy); 14.583 oz. (troy); 16 oz. (avoir.); 291.667 pennyweights; 7000 grains; 453.592 grams.
Pound (troy) (lb. tr.)	0.82286 lb. (avoir.); 12 oz. (troy); 13.166 oz. (avoir.); 240 pennyweights; 5760 grains; 373.24 grams.
Quart (qt.) (liquid)	0.03342 cubic foot; 0.25 gallon; 32 oz. (fluid); 57.749 cubic inches; 946.358 cubic centimeters.
Square Centimeter ($cm.^2$)	0.0010764 sq. ft.; 0.155 sq. in.
Square Decimeter ($dm.^2$)	15.50 sq. in.; 0.01 sq. meter; 100 sq. centimeters.

Square Foot ($ft.^2$)	144 sq. in.; 929.034 sq. centimeters.
Square Inch ($in.^2$)	0.006944 sq. ft.; 6.4516 sq. centimeters.
Square Meter ($m.^2$)	1.196 sq. yd.; 10.7639 sq. ft.; 1550.0 sq. in.; 10,000 sq. centimeters.

CONVERSION FACTORS

Grams per liter (g./l.) multiplied by 0.134=avoirdupois ounces per gallon (oz./gal.).

Avoirdupois ounces per gallon (oz./gal.) multiplied by 7.5=grams per liter (g./l.).

Grams per liter (g./l.) multiplied by 0.122=troy ounces per gallon (tr. oz./gal.).

Troy ounces per gallon (tr. oz./gal.) multiplied by 8.2=grams per liter (g./l.).

Grams per liter (g./l.) multiplied by 2.44=pennyweights per gallon (dwt./gal.).

Pennyweights per gallon (dwt./gal.) multiplied by 0.41=grams per liter (g./l.).

Amperes per square decimeter ($amp./dm.^2$) multiplied by 9.29=amperes per sq. ft. ($amp./ft.^2$).

Amperes per square foot ($amp./ft.^2$) multiplied by 0.108=amperes per square decimeter ($amp./dm.^2$).

Fluid ounces per gallon (fl. oz./gal.) multiplied by 7.7=cubic centimeters per liter (c.c./l.).

COMMON pH INDICATORS

Common Name	*pH Range*	*Color Change*
Acid cresol red	0.2 — 1.8	red — yellow
Meta cresol purple (acid range)	1.2 — 2.8	red — yellow
Thymol blue (acid range)	1.2 — 2.8	red — yellow
Brom phenol blue	3.0 — 4.6	yellow — purple
Brom cresol green	3.8 — 5.4	yellow — blue
Methyl red	4.2 — 6.2	red — yellow
Chlor phenol red	4.8 — 6.4	yellow — red
Brom phenol red	5.2 — 6.8	yellow — red
Brom cresol purple	5.2 — 6.8	yellow — purple
Brom thymol blue	6.0 — 7.6	yellow — blue
Phenol red	6.4 — 8.0	yellow — red
Cresol red	7.2 — 8.8	yellow — red
Meta cresol purple (alkaline range)	7.6 — 9.2	yellow — purple
Thymol blue (alkaline range)	8.0 — 9.6	yellow — blue
Phthalein red	8.6 — 10.2	yellow — red
LaMotte purple	9.6 — 11.2	purple — red purple
Tolyl red	10.0 — 11.6	red — yellow
Alizarin yellow	10.1 — 12.0	yellow — violet
LaMotte sulfo orange	11.0 — 12.6	dark orange — light orange
Parazo orange	11.0 — 12.6	yellow — orange
LaMotte violet	12.0 — 13.6	violet — blue green
Acyl blue	12.0 — 13.6	red — blue

METAL CONTENT OF COMMON PLATING SALTS

By Nathan E. Promisel
Washington, D. C.

(**Note:** Technical products usually vary slightly from stated percentages.)

Technical Name of Salt	Chemical Formula	Per Cent Metal
Antimony trichloride	$SbCl_3$	53.4
Cadmium cyanide	$Cd(CN)_2$	68.3
Cadmium oxide	CdO	87.5
Chromic acid	CrO_3	52.0
Cobalt sulfate (anhydrous)	$CoSO_4$	38.0
Cobalt sulfate, crystal	$CoSO_4 \cdot 7H_2O$	21.0
Copper carbonate (basic)	$CuCO_3 \cdot Cu(OH)_2$	57.5
Copper chloride (ic)	$CuCl_2$	47.3
Copper cyanide (ous)	$CuCN$	71.0
Copper sulfate (ic), crystal	$CuSO_4 \cdot 5H_2O$	25.5
Ferrous chloride, crystal	$FeCl_2 \cdot 4H_2O$	28.1
Ferrous ammonium sulfate	$FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$	14.2
Gold chloride (ic)	$AuCl_3$	64.9
Gold chloride (ic), crystals	$AuCl_3 \cdot 2H_2O$	58.1
Gold chloride (ous)	$AuCl$	84.7
Gold cyanide (ous)	$AuCN$	88.3
Gold potassium cyanide	$KAu(CN)_2$	68.3*
Gold potassium cyanide, crystal	$KAu(CN)_2 \cdot 2H_2O$	60.8
Gold sodium cyanide	$NaAu(CN)_2$	72.5*
Lead carbonate (basic)	$Pb(OH)_2 \cdot 2PbCO_3$	80.1
Mercuric chloride	$HgCl_2$	73.8
Mercuric cyanide	$Hg(CN)_2$	79.4
Mercuric nitrate	$Hg(NO_3)_2$	61.8
Mercuric nitrate, crystal	$Hg(NO_3)_2 \cdot H_2O$	58.6
Nickel ammonium sulfate (double nickel salts)	$NiSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$	14.9
Nickel carbonate (basic)	$2NiCO_3 \cdot 3Ni(OH)_2 \cdot 4H_2O$	50.0
Nickel chloride, crystal	$NiCl_2 \cdot 6H_2O$	24.7
Nickel sulfate (single nickel salt)	$NiSO_4 \cdot 6H_2O$	22.3
Platinum chloride, crystal	$H_2PtCl_6 \cdot 6H_2O$	37.7
Rhodium phosphate, crystal	$RhPO_4 \cdot 3H_2O$	29.9
Rhodium sulfate, crystal	$Rh_2(SO_4)_3 \cdot 12H_2O$	29.0
Silver chloride	$AgCl$	75.3
Silver cyanide	$AgCN$	80.5
Silver potassium cyanide	$KAg(CN)_2$	54.2
Silver sodium cyanide	$NaAg(CN)_2$	59.0
Silver nitrate	$AgNO_3$	63.5
Sodium stannate, crystal	$Na_2SnO_3 \cdot 3H_2O$	44.5
Tin chloride (ous), crystal	$SnCl_2 \cdot 2H_2O$	52.6
Tin sulfate (ous)	$SnSO_4$	55.3
Tungstic acid	H_2WO_4	73.6
Tungstic oxide	WO_3	79.3
Zinc cyanide	$Zn(CN)_2$	55.7
Zinc oxide	ZnO	80.3
Zinc sulfate, crystal	$ZnSO_4 \cdot 7H_2O$	22.7

*Technically salts may contain appreciably less, and gold content is usually stated on container.



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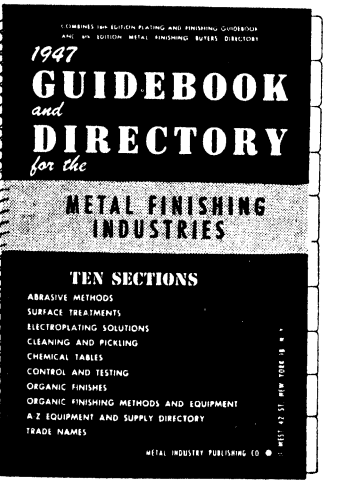
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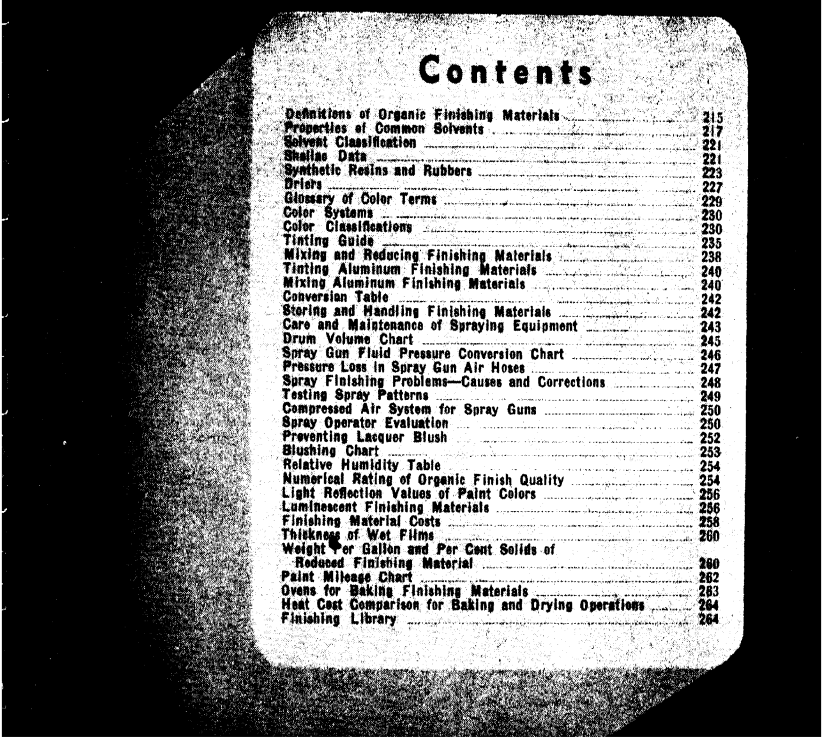


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ORGANIC FINISHING

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ORGANIC FINISHING

WILLOUGHBY G. SHEANE, Editor

DEFINITIONS OF ORGANIC FINISHING MATERIALS

PAINT. An opaque, colored combination of drying oil (linseed oil, perilla oil, etc.) and suitable pigments and solvents, with or without driers, resins, etc., used mainly for architectural purposes. Paints dry relatively slowly and cure at ordinary temperatures.

LACQUER. A combination of cellulose ester (cellulose nitrate, cellulose acetate) and one or more plasticisers, resins and solvents, with or without pigments, etc., used principally in industrial finishing. Due to the nature of the solvents employed, drying is rapid, even under ordinary conditions. Curing is accomplished by air-drying at room temperature or by "force-drying" at slightly elevated temperatures.

Lacquer Dope. A special type of lacquer of cellulose nitrate or cellulose acetate base used for coating airplane fabric, leather, paper, etc. Also, any stock cellulose ester solution used as a base for further formulation.

Pyroxylin Lacquer. A lacquer employing a cellulose nitrate (nitrocellulose) base.

Clear Lacquer. A transparent to translucent lacquer, either colorless or colored, whose formulation includes no pigments or other opacifying agents.

Pigmented Lacquer. Any opaque, colored lacquer.

VARNISH. A usually unpigmented, colorless combination of resin and drying oil, solvents and, generally, driers. Varnishes find most application in architectural finishing, although some are designed for industrial purposes. Drying takes place slowly at room temperature. Curing may be accomplished with or without heat, depending on the formulation.

Long Oil Varnish. A varnish in which the proportion of drying oil to resin is large. In general, long oil varnishes require a somewhat longer period in which to cure and are more flexible and durable than those of the short oil variety

Spar Varnish. A long oil varnish of high grade, designed for outdoor use and where durability is required.

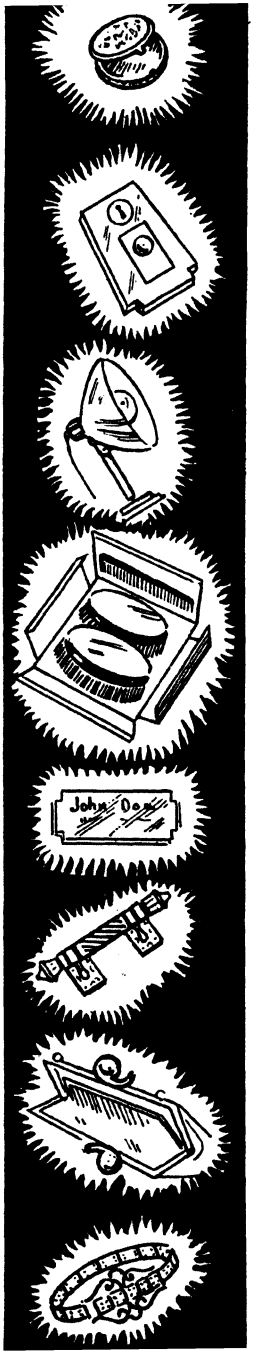
Color Varnish. A varnish in which pigments have been incorporated to produce a desired color.

Spirit Varnish. A finishing material similar in characteristics to varnish, but usually made without heat by simple solution of resins in solvents, especially alcohol.

ENAMEL. A pigmented combination employing either a resin or varnish base. Enamels are used for both industrial and architectural purposes. They dry somewhat more rapidly than paints and less rapidly than lacquers. Depending on their formulation, they may be cured at room temperature or at elevated temperatures.

Lacquer Enamel. An enamel employing a lacquer base.

JAPAN. A dark colored and usually black combination of a bituminous material such as asphalt, drying oils and solvents, with or without pigments, driers, etc., used principally for industrial purposes. Depending on their composition,



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japans are cured by baking at elevated temperatures or by air-drying at ordinary temperatures.

Water Japan. A japan so prepared as to use water in place of the usual organic solvents.

SHELLAC. The alcohol solution of the natural resin shellac, with or without oils, other resins, etc. Shellac dries quickly and its use is generally limited to indoor architectural applications.

SYNTHETIC. Any organic finishing material whose formulation is based on synthetic, i.e. manufactured, resins rather than those of natural origin.

BITUMINOUS PAINT, ENAMEL, etc. Any of the usual types of finishing materials which employs appreciable amounts of bituminous substances in its formulation. Bituminous finishing materials are of necessity limited to dark colors.

PROPERTIES OF COMMON SOLVENTS

Solvent	Boiling Range °C.	Flash Point (Open Cup) °F.	Wt./Gal. at 20°C. lbs.
ALCOHOLS			
Methyl Alcohol	64- 66	52	6.60
Ethyl Alcohol	74- 79	65	6.58
Isopropyl Alcohol	82- 83	67	6.54
Secondary Butyl Alcohol	96-103	88	6.72
Isobutyl Alcohol	107-111	111	6.68
Normal Butyl Alcohol	116-119	111	6.75
Secondary Amyl Alcohol	117-124	110	6.74
Amyl Alcohol (mixed isomers)	121-139	111	6.77
Methyl Amyl Alcohol	131-132	114	6.72
Hexyl Alcohol	144-156	145	6.93
Octyl Alcohol	182-202	178	6.94
ESTERS			
Methyl Acetate (82%)	53- 55	5	7.55
Ethyl Acetate (85%)	70- 80	35	7.37
Isopropyl Acetate (85%)	82- 90	45	7.13
Isopropyl Acetate	84- 94	35	7.23
Diethyl Carbonate (91%)	87-127	85	7.96
Secondary Butyl Acetate (82%)	105-127	88	7.14
Isobutyl Acetate	114-118	88	7.24
Normal Butyl Acetate (90%)	119-127	88	7.29
Ethyl Lactate	119-176	140	8.60
Secondary Amyl Acetate (92%)	121-144	111	7.18
Normal Butyl Propionate	124-171	105	7.27
Amyl Acetate (mixed isomers) (84%)	127-155	111	7.17
Methyl Amyl Acetate	140-147	113	7.13
Methyl Cellosolve Acetate	143-145	132	8.37
Cellosolve Acetate	145-166	122	8.10



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Isopropyl Lactate	149-167	130	8.22
Hexyl Acetate (91%)	157-164	135	7.29
Butyl Cellosolve Acetate	188-192	180	7.85
Octyl Acetate	195-203	180	7.27
Methyl Carbitol Acetate	203-212	180	8.65
Carbitol Acetate	211-220	225	8.41
Butyl Carbitol Acetate	236-249	260	8.21
ETHER ALCOHOLS			
Methyl Cellosolve	121-126	105	8.04
Cellosolve	133-137	111	7.75
Isopropyl Cellosolve	140-143	125	7.53
Butyl Cellosolve	163-172	165	7.50
Methyl Carbitol	190-194	200	8.62
Carbitol	189-203	205	8.55
Butyl Carbitol	220-231	230	7.95
Benzyl Cellosolve	254-258	265	8.90
KETONES			
Acetone	55- 57	0	6.60
Methyl Ethyl Ketone	77- 82	34	6.73
Diethyl Ketone	100-104	59	6.78
Methyl Propyl Ketone	101-107	60	6.74
Methyl Isobutyl Ketone	112-118	74	6.67
Mesityl Oxide (86%)	117-139	90	7.12
Dipropyl Ketone	138-144	120	6.79
Methyl Normal Amyl Ketone	147-153	115	6.81
Diacetone Alcohol	153-160	135	7.79
Diisobutyl Ketone	164-169	145	6.72
Acetonylacetone	188-193	190	8.10
HYDROCARBONS			
Benzene	78- 81	<5.5	7.31
Petroleum Naphtha	96-129	31	6.11
Toluene	109-111	44	7.21
Xylene	127-159	80	7.17
Hi-Flash Naphtha	149-193	115	7.16
Mineral Spirits	152-207	140	6.40
V. M. and P. Naphtha	168-210	120	6.36
MISCELLANEOUS			
Ethylene Dichloride	82- 84	70	10.45
Turpentine (Gum Spirits)	154-201	110	7.22
Turpentine (Steam Distilled)	155-173	115	7.13

Except where noted, alcohols, esters, ether alcohols and ketones are 95% or greater purity. Data based on "Lacquer Solvents in Commercial Use," Doolittle, Ind. & Eng. Chem., **27**, No. 10.

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SOLVENT CLASSIFICATION

Solvents are often classified as "low," "medium" or "high" boilers, that is, they are grouped according to their boiling points. This system is not exact, of course, and there are many instances where overlapping occurs.

Given below are the various classes, with examples of the types of solvents commonly used in finishing materials.

Class I. Low Boilers. Boiling below 100° C.

Type	*Example*	*Boiling Range—°C*
Alcohol	Ethyl Alcohol	74-79
Ester	Ethyl Acetate (85%)	70-80
Ketone	Acetone	55-57
Coal Tar Hydrocarbon	Benzene	78-81

Class II. Medium Boilers. Boiling near 125° C.

Type	*Example*	*Boiling Range—°C.*
Alcohol	Normal Butyl Alcohol	116-119
Ester	Ethyl Butyrate	107-131
Ketone	Methyl Isobutyl Ketone	112-118
Ether-Alcohol	Methyl Cellosolve	121-126
Coal Tar Hydrocarbon	Toluene	109-111
Petroleum Hydrocarbon	Petroleum Naphtha	96-129

Class III. High Boilers. Boiling between 150° and 200° C.

Type	*Example*	*Boiling Range—°C.*
Alcohol	Benzyl Alcohol	199-204
Ester	Butyl Lactate	145-230
Ketone	Acetonylacetone	188-193
Ether-Alcohol	Methyl Carbitol	190-194
Coal Tar Hydrocarbon	Xylene	127-159
Petroleum Hydrocarbon	VM&P Naphtha	152-207

SHELLAC DATA

Cut (lbs. dry shellac per gal. alcohol)	Wt./Gal. (lbs.)	Per Cent Solids	Alcohol Content (gals./gal. shellac)
1.0	7.1	12.9	0.91
1.5	7.2	18.1	0.87
2.0	7.3	22.8	0.83
2.5	7.4	26.9	0.80
3.0	7.5	30.6	0.77
3.5	7.6	33.8	0.74
4.0	7.7	37.0	0.71
4.5	7.8	39.7	0.69
5.0	7.9	42.4	0.67

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RIDOSOL improves the detergent properties of alkali cleaning solutions—especially useful when solutions are used at lower temperatures.

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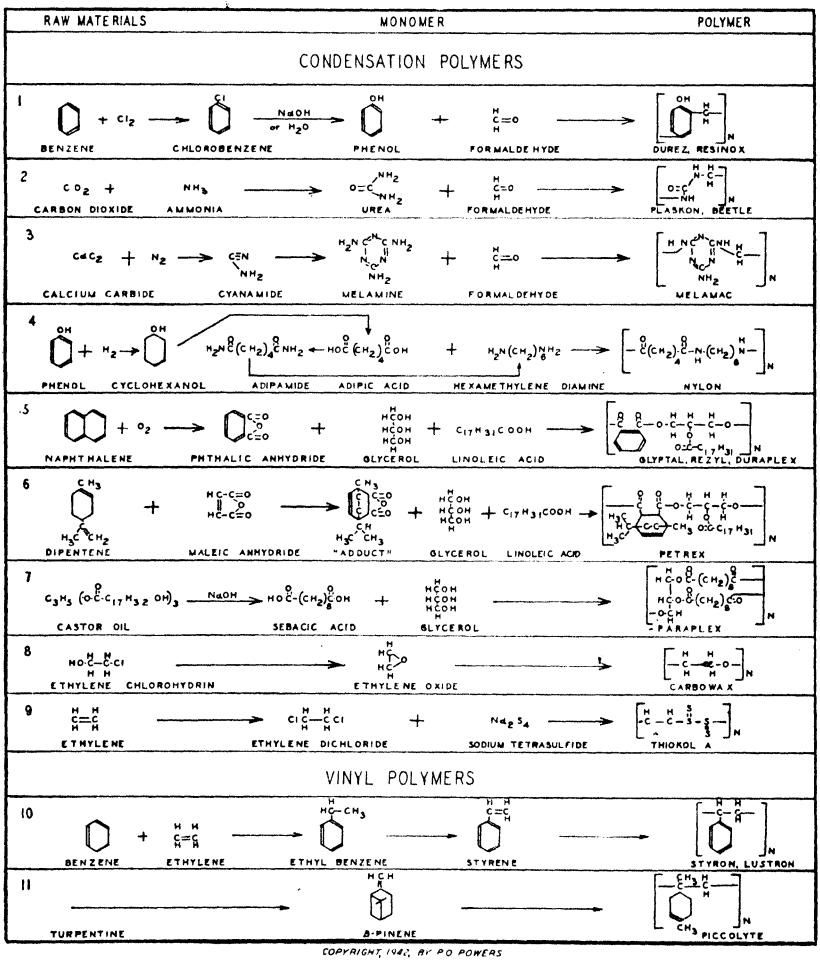
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SYNTHETIC RESINS AND RUBBERS

By P. O. Powers

This chart lists important resins and rubbers produced in commercial quantities in the United States. Some of the registered trade marks or trade names designating the resins are used. For brevity, no attempt has been made to include all of these nor company trade marks ordinarily associated with more than one type of resin. The formulas for the resins are necessarily abbreviated and may not always be entirely accurate. In some cases the formulas used have not been previously suggested.



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(Continued on page 225)

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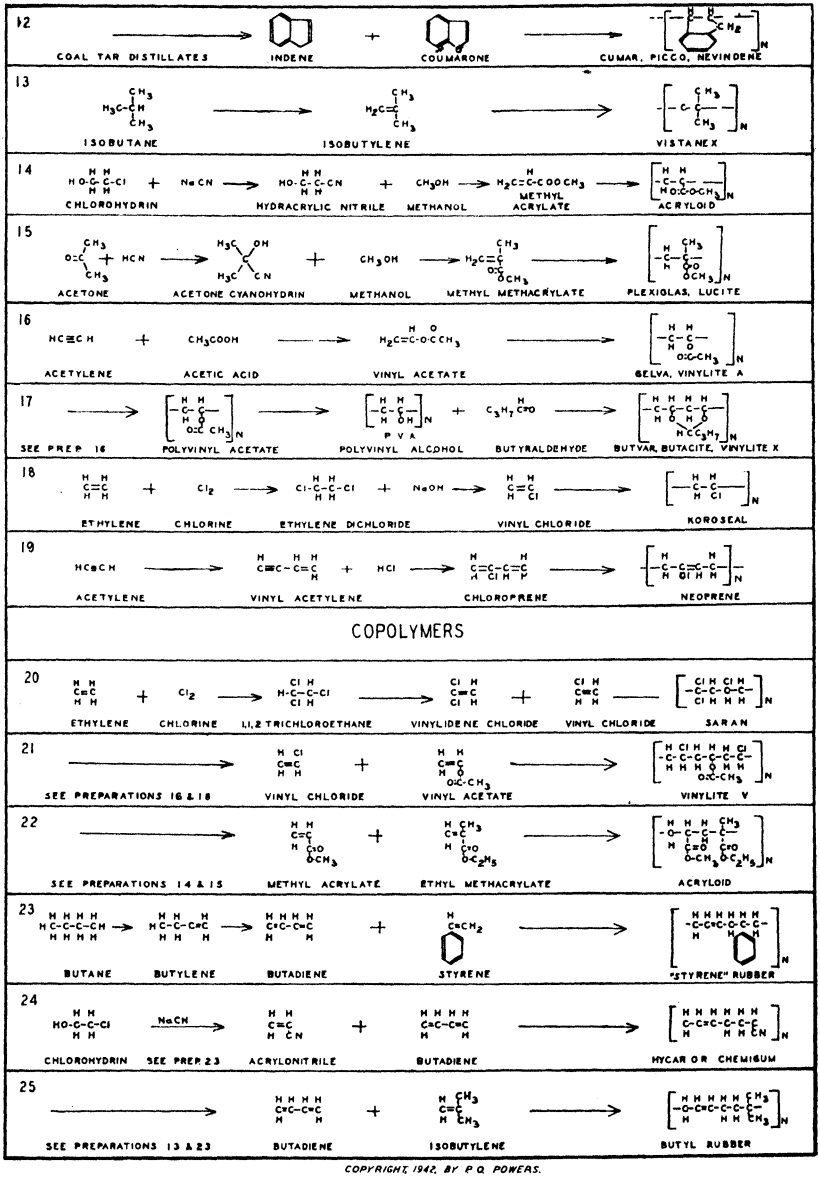
Air dry crystal lacquer.

★

Baking texture enamel.

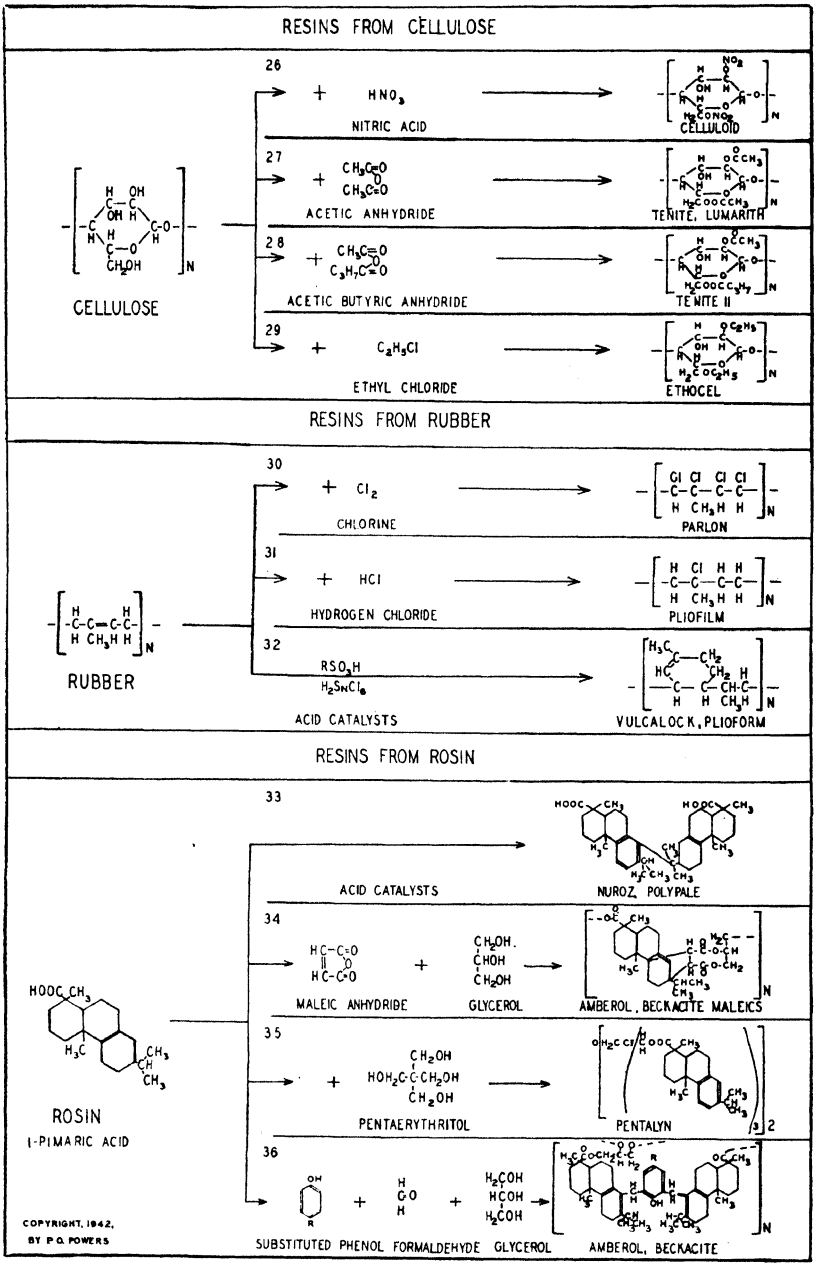
Randolph Finishing Products Company

Carlstadt, New Jersey



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(Continued on page 226)


RESINS FROM CELLULOSE
CELLULOSE
26
+ HNO3
NITRIC ACID
CELLULOID
27
+ CH3C=O / CH3C=O
ACETIC ANHYDRIDE
TENITE, LUMARITH
28
+ CH3C=O / C3H7C=O
ACETIC BUTYRIC ANHYDRIDE
TENITE II
29
+ C2H5Cl
ETHYL CHLORIDE
ETHOCEL
RESINS FROM RUBBER
RUBBER
30
+ Cl2
CHLORINE
PARLON
31
+ HCl
HYDROGEN CHLORIDE
PLIOFILM
32
RSO3H
H2SnCl6
ACID CATALYSTS
VULCALOCK, PLIOFORM
RESINS FROM ROSIN
ROSIN
I-PIMARIC ACID
33
ACID CATALYSTS
NUROZ, POLYPALE
34
+
MALEIC ANHYDRIDE
GLYCEROL
AMBEROL, BECKACITE MALEICS
35
+
PENTAERYTHRITOL
PENTALYN
36
+ +
SUBSTITUTED PHENOL FORMALDEHYDE GLYCEROL
AMBEROL, BECKACITE
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DRIERS

Driers, although present in relatively small amounts, are perhaps the most important ingredients which the coating manufacturer must use so that the finisher can be supplied with materials to meet his requirements.

The following information is offered to provide an understanding and appreciation of the function and mechanism of driers. It is not to be construed as instructions for their use. Directions of finishing material suppliers must be followed exactly at all times if satisfactory results are to be obtained.

* * *

Paints, varnishes and other finishing materials of similar type cannot be utilized to the fullest unless the proper type and quantity of drier is used. Inasmuch as these finishing materials in the wet state have no value until they are converted to a dry film, driers are essential because without them this conversion takes too long for practical purposes. Therefore, driers should be considered as catalysts which, when added to finishing materials, accelerate the formation of a useful film by increasing the speed of oxidation and/or polymerization and/or gelation.

Effect of Drying Conditions

(a) *Temperature*—The effectiveness of driers increases rapidly with an increase in temperature. Therefore, the amount of driers must be adjusted to the temperatures involved.

(b) *Humidity*—As humidity increases, the effectiveness of driers is generally decreased.

(c) *Light*—In the presence of light the effectiveness of driers is much more marked. Paints dry much slower in the dark.

(d) *Ventilation*—A supply of fresh air removes evaporating solvents from the wet film and brings to it large volumes of oxygen. Hence, good ventilation is essential.

(e) *Film Thickness*—Finishing materials have an optimum thickness at which they dry properly. Thick films tend to top dry or wrinkle.

Function of Driers

Given below are the functions of the principal driers employed in the finishing industry.

It should be emphasized that the individual driers listed are seldom used alone but rather in combinations of two or more. The formulator must consider the functions of each, as well as the type finish he is preparing in order to select the proper drier combination.

Cobalt Drier—This is the most active drier, sometimes used alone but more often with an auxiliary drier such as lead, calcium or zinc. This drier is often referred to as a "top" drier because it has the tendency, particularly when used in excess, to dry the surface of the protective film too rapidly. It is essential for finishing materials known to be applied under humid conditions.

Lead Drier—This is the most important auxiliary drier. It is rarely used alone but is usually combined with cobalt or manganese, or both, to give balanced drying. High lead driers offset the tendency of thick films to top dry or wrinkle.

Manganese Drier—This drier is used in air drying finishes in conjunction with lead drier. It is often used in baking finishes where it is usually very effective even at low concentrations.

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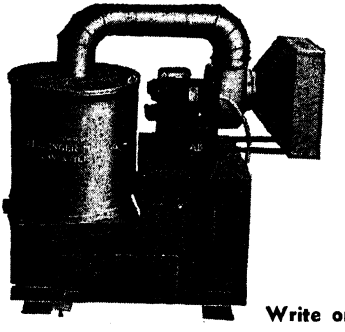
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Zinc Drier— This drier is considered a mild auxiliary drier and is usually used with cobalt as it inhibits the excessive top drying of such an active drier as cobalt. In this manner it prevents wrinkling. Zinc drier is also used to replace lead where lead cannot be employed due to its toxicity or due to its tendency to discolor in the presence of sulfides.

Iron Drier—This unique drier is of value when used in conjunction with cobalt and manganese in certain types of air dried finishing materials. It is becoming more popular in baked finishing materials and may be used alone. Due to staining, it is limited to black and colors and tints other than white. This drier is often very effective in reducing surface tack of paints containing fish oil.

Calcium Drier—This drier is of the auxiliary type such as lead and zinc. It has several valuable applications. It can be used to replace lead where this metal cannot be used. When used in conjunction with lead it serves to stabilize the driers in those vehicles that have a tendency to form a "lead haze." In baking finishes, calcium often shows better performance as an auxiliary drier than lead or zinc.

GLOSSARY OF COLOR TERMS

Black. The absence of color. Zero brilliance and 100% saturation.

Brilliance. The percentage of incident light reflected by an object. A perfectly white material has 100% brilliance while an absolute black has zero brilliance.

Chromatic Scale. The colors of the spectrum—red, orange, yellow, green, blue, violet.

Color. That quality by which bodies have a different appearance, independently of their form, depending on the effect of different wave lengths on the retina of the eye.

Color Scale. A series of colors grading from one hue to another or a similar series of tones of one hue.

Complementary Colors. Colors which together make white. Red and green, blue and orange, yellow and violet or complementary pairs of colors.

Hue. The predominant wave length reflected by a colored material which determines its position in spectral or chromatic scale. It is an attribute of a masstone shade or tint.

Masstone. A full strength color. The hue of a color not toned with white or black.

Pastel. A tint. A masstone to which white has been added.

Pigment. An insoluble material that imparts a color or black or white to the material to which it is added.

Primary Colors. Red, yellow and blue.

Saturation. The percentage of reflected light which is colored.

Secondary Colors. Orange, green and violet. Mixtures of primary colors. Orange from a blend of red and yellow; green from yellow and blue; violet from blue and red.

Shade. A color toned with black or with a complementary color having some degree of dulling effect.

Spectrum. The series of hues of continuous gradation of which white light is composed.

Tertiary Colors. Citron, olive and russet. Mixtures of secondary colors. Citron

from a blend of orange and green; olive from green and violet; russet from violet and orange.

Tint. A masstone color to which white has been added. A pastel color is a tint.

Tone. A step in the scale of a single color or hue, ranging from the lightest tint to the darkest shade of that color.

White. Zero saturation and 100% brilliance.

COLOR CLASSIFICATIONS

The spectrum has been arbitrarily divided into six broad regions designated as violet, blue, green, yellow, orange and red. Since the appearance of the spectrum varies continuously throughout its length, this classification of spectral colors is only approximate. A more precise classification is provided by the following scale based on wave length.

Color	*Wavelength Range (Millimicrons)*
Violet	400 to 460
Blue	460 to 500
Green	500 to 570
Yellow	570 to 590
Orange	590 to 610
Red	610 to 700

Colors are also classified as primary, secondary and tertiary.

The primary colors are red, yellow and blue.

The secondary colors are orange, green and violet and may be obtained by mixing pairs of primary colors as follows:

Red and yellow make orange.
Yellow and blue make green.
Blue and red make violet.

The tertiary colors are citron, olive and russet and may be obtained by mixing pairs of secondary colors as follows:

Orange and green make citron.
Green and violet make olive.
Violet and orange make russet.

COLOR SYSTEMS

There are two major systems of color which may lay claim to wide recognition both in America and Europe. These are the creations of Albert H. Munsell (1859-1918) and Wilhelm-Ostwald (1853-1932).

The two systems are greatly alike in several respects:

1. An orderly sequence of colors which plot the entire realm of visual sensation.

2. A three-dimensional conception in which the general form of the solid comprises a white and a black apex, a neutral gray axis, pure hues about an

equator and intermediate scales of tints, shades and tones in methodical arrangement.

3. A method of color identification in which each individual tone within the solid is to be measured and noted.

MUNSELL SYSTEM

Basically, the Munsell system follows the general shape of a sphere. However, because all pure colors do not have like brightness or purity, light hues like yellow are placed near the white apex and dark colors like purple near the black. Again, colors of strong saturation, like red, extend further from the neutral gray axis than colors like blue-green. These features are illustrated in Figures 1, 2 and 3.

In the Munsell system the three dimensions of color are *hue*, *value* and *chroma*.

Hue is the quality which distinguishes red from orange, orange from yellow, etc. It is identified in the system with letters as follows:

R: Red	BG: Blue-Green
YR: Yellow-Red	B: Blue
Y: Yellow	PB: Purple-Blue
GY: Green-Yellow	P: Purple
G: Green	RP: Red-Purple

Value refers to apparent lightness as compared with white, grays or black. Nine value steps from black to white have numbers from 1 through 9.

Chroma refers to purity, saturation or apparent degree of departure from a "neutral" gray. A hue of low chroma will thus have a low number (1, 2, 3). Hues of strong chroma will have correspondingly larger numbers, a brilliant red reaching a chroma of 14 or more.

Hence, hue is given a symbolic letter. The identification of value follows next with a number and the identification of chroma is third with another number.

Examples of Munsell Notations

Y 3/2. This would be a deep olive color, being a yellow equivalent in value

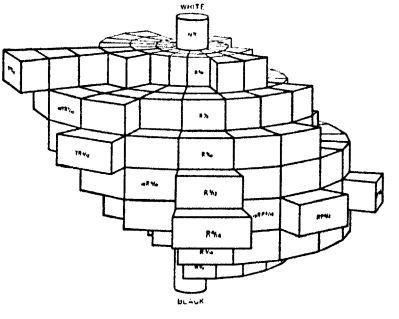


Figure 1. Munsell color solid. Light colors appear at a level near white and dark colors at a level near black. Intense hues extend further from the gray scale than pale colors.

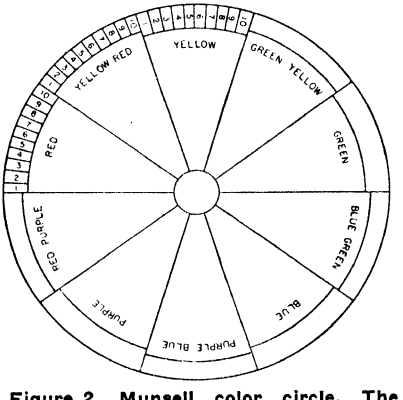


Figure 2. Munsell color circle. The complete circle contains 100 hues.

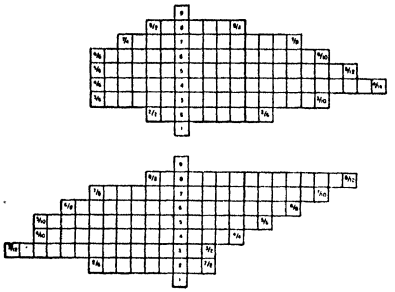


Figure 3. Sections of Munsell color solid. These are vertical sections. Numbers from 1 through 9 indicate the gray scale. Upper diagram shows tone variations and scales of red and blue-green. Lower diagram shows scales of yellow and purple-blue.

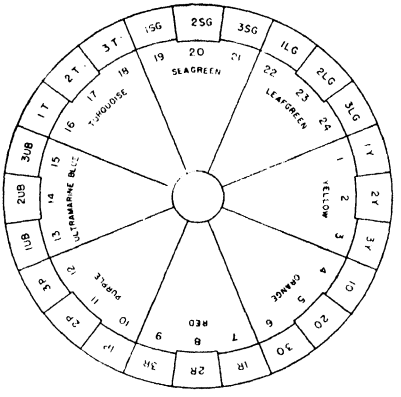


Figure 5. Ostwald color circle. The complete circle contains 24 hues with red, yellow, sea green and ultramarine blue (the psychological primaries) as a basis.

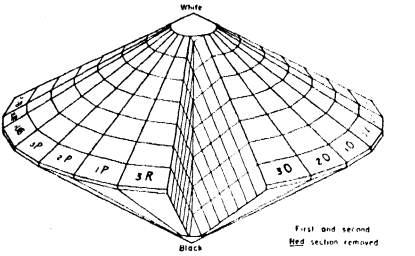


Figure 4. Ostwald color solid. Pure hues extend about the equator. These form graded scales toward white, black and gray.

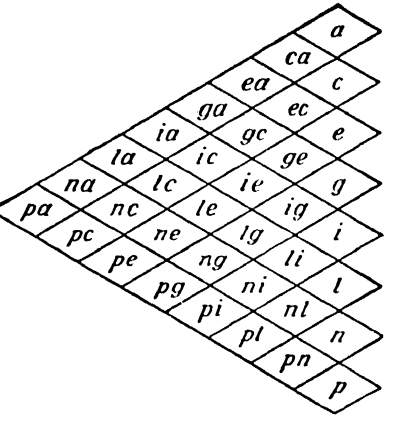


Figure 6. Section of Ostwald color solid. Letters a through p indicate the gray scale. The angle pa is pure color. All other tones comprise mixtures of white and black with hue—the first letter representing the white content and the second letter the black. Scales running in oblique directions have equal white or equal black contents.

to a deep gray (3) and weak in chroma (only 2 steps from neutrality).

PB 7/6. This would be a lavender (purple-blue). The value (7) is equivalent to a fairly light gray. The chroma, 6 steps from neutrality, is clear and bright.

R 5/3. This would be a soft grayish red midway in brightness between black and white and only three steps removed from gray.

OSTWALD SYSTEM

Whereas the Munsell system finds its dimensions in hue, value and chroma (which correspond to dominant wave length, luminous apparent reflectance and colorimetric purity, as recognized in most colorimetric practice), Ostwald is concerned with *hue, black* and *white.* Following principles stated by psychologists

such as Ewald Hering, Ostwald's system recognizes that every color seen by the eye is composed of proportions of hue, of white and black and that all such elements are measurable. His solid, as illustrated in Figure 4, has the form of a double cone. His color circle (Figure 5), at the circumference, is made up of 24 hues which are designated with numbers, as follows:

1:	13:
2: Yellow	14: Ultramarine
3:	15: Blue
4:	16:
5: Orange	17: Turquoise
6:	18:
7:	19:
8: Red	20: Sea Green
9:	21:
10:	22:
11: Purple	23: Leaf Green
12:	24:

Each of the 24 hues is made to comprise a monochromatic triangle having 28 tones. The neutral gray scale has 8 steps and is identified with the letters *a* (for white), *c, e, g, i, l, n, p* (for black).

Every color, pure or modified, is to be described with a combination of these letters. And the letters indicate the proportionate amounts of white and black contained in the tone.

Examples of Ostwald Notations

The numeral refers to the hue as on the color circle. The first letter refers to the white content and the second letter to the black content—both traced from corresponding amounts of white or black on the gray scale.

8 *pa.* This would be a relatively pure red. The first letter *p* (black) indicating no white in the mixture, the second letter *a* (white) indicating no black.

2 *pi.* This would be a deep yellow or olive, the first letter *p* indicating an absence of white, the second letter *i* a slight amount of black.

11 *ga.* This would be a lavender with a *g* amount of white, as on the gray scale, and no black.

8 *le.* This would be a soft grayish red having an *l* amount of white and an *e* amount of black, as on the gray scale.

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TINTING GUIDE

Add tinting colors in small quantities and with thorough stirring until desired color is obtained. Compiled by National Paint, Varnish and Lacquer Assn.

Cream—to white add raw sienna, varying the amount to obtain pale, light or dark cream as desired.

Grayed Cream—to cream add a trace of lampblack.

Ivory—to white add a very little raw sienna and a trace of vermilion, or a little yellow ochre and a trace of venetian red.

Pale Tan—to white add a small amount of burnt umber.

Creamy Tan—to pale tan add a little raw sienna.

Ecru—to white add medium chrome yellow and a little toluidine red.

Buff—to white add an appreciable amount of raw sienna.

Maize—to white add a considerable amount of medium chrome yellow and some lemon chrome yellow.

Sand—to white add yellow ochre and a trace of black and vermilion.

Fawn—to white add burnt umber and yellow ochre.

Pale Yellow—to white add a small amount of chrome yellow or medium chrome yellow.

Colonial Yellow—to white add French ochre and medium chrome yellow. Use approximately twice as much ochre as chrome.

Dull Gold—to white add considerable light chrome yellow and a little venetian red.

Orange Yellow—to white add medium chrome yellow and a very little vermilion.

Pale Orange—to white add a small amount of vermilion and chrome yellow.

Burnt Orange—to white add vermilion and chrome yellow, graying off with a trace of Prussian Blue.

Topaz—to white add orange chrome yellow or medium chrome yellow and a little vermilion.

Peach—to white add chrome yellow and a little vermilion.

Pink—to white add a small amount of vermilion.

Rose—to white add vermilion, turkey red or rose lake until the desired shade is produced.

Salmon—to white add considerable deep orange chrome, graying off slightly with a trace of Prussian blue if desired.

Coral—to white add medium chrome yellow and a little vermilion.

Brick Red—to white add considerable venetian red and about half as much Indian red.

Dark Red—to brick red add a bit of lampblack.

Maroon—to white and venetian red, a little black and a trace of Prussian blue.

Reddish-Brown—to burnt umber material add venetian red. Add white if a lighter brown is desired.

Light Yellowish-Brown—to white add chrome yellow and burnt umber.

Cinnamon Brown—to white add raw sienna and golden ochre, more sienna than ochre.

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Green—to white add medium chrome green and medium chrome yellow in varying amounts to obtain desired depth of color.

Soft Green—to white add chrome yellow, chrome green and raw sienna.

Pale Green—to white add very small amounts of medium chrome green and medium chrome yellow. Deeper green may be obtained by using larger quantities of the same colors.

Apple Green—to white add light chrome green and orange chrome.

Sea Green—to white add deep chrome green.

Adam Green—to white add medium chrome green and a little Chinese blue.

Jade Green—to white add considerable chrome green, a little Prussian blue and a little venetian red.

Yellow-Green—to white add a little medium chrome green and a touch of medium chrome yellow, varying the yellow to obtain the desired shade.

Soft Yellowish-Green—to white add medium chrome green, raw sienna and a trace of medium chrome yellow.

Olive Green—to white add chrome yellow and lampblack.

Nile Green—to white add Prussian blue and emerald green, about three times as much color as white.

Gray-Green—to white add varying amounts of chrome yellow and chrome green, graying off with a very little venetian red.

Blue-Green—to white add Prussian blue and chrome green in varying quantities to produce light, medium or dark as desired.

Pale Blue—to white add a very little Prussian blue. Deeper shades require more Prussian blue.

Delft Blue—to white add cobalt blue and a touch of lampblack.

Gray-Blue—to white add Prussian blue and lampblack. For a livelier color, replace the lampblack with orange chrome.

Gray—to white add lampblack until the desired shade is obtained.

Silver Gray—to white add a trace of lampblack and raw umber and, if preferred, a trace of raw sienna.

Pearl Gray—to white add chromium oxide green and red iron oxide.

Stone Gray—to white add raw sienna and raw umber.

French Gray—to gray add Prussian blue.

Stone Gray—to white add raw sienna and raw umber.

Limestone Gray—to white add raw sienna and raw umber.

Mauve-Gray—to white add rose lake and some cobalt blue, graying off with a trace of lampblack.

Pale Lavender—to white add about three parts of ultramarine blue and one part of madder lake.

Grayed Lavender—to white add ultramarine blue and madder lake, graying off with a little chrome yellow.

Lilac—to white add a small amount of ultramarine blue and rose madder.

Orchid—to white add rose pink. Wisteria and lavender, also basically violet-red, can be made the same way.

MIXING AND REDUCING FINISHING MATERIALS

Many of the troubles and unsatisfactory results obtained with finishing materials, particularly of the pigmented variety, can be traced to improper mixing and reducing. If materials are not mixed and reduced properly, differences in color, gloss, hiding power, film and application characteristics may be expected.

1. Hand mixing in cans. Where hand mixing in cans must be used, the method illustrated and described in Figure 1 will be found effective.

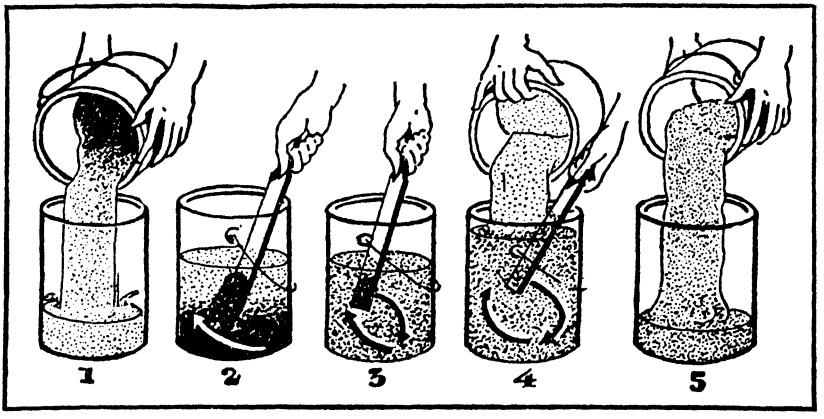


Fig. 1. Hand Mixing in cans.

1. Pour a quarter of contents into another clean can.
2. Loosen any settled pigment with a wide paddle.
3. Mix thoroughly, using a lifting and beating motion.
4. Gradually add material poured off, mixing as in step 3.
5. Complete mixing by pouring back and forth from one can into another (boxing).

2. Hand mixing in open head drums. If a mule for rotating drums or electric- or air-driven agitator are not available, remove head of drum and mix as described in Figure 2. Do not attempt to mix through the bung hole. It cannot be done. Use a strong paddle, four or five inches wide and two or three feet longer than the drum. Mixing can be facilitated by removing a quarter of the material, mixing thoroughly and then adding the part drawn off as suggested for mixing in cans.

3. Mixing in drums equipped with built-in agitators. Many drums are equipped with built-in agitating devices. These may be operated by hand or by electric- or air-driven motors.

4. Reducing finishing materials. Reducer should be added slowly and with vigorous stirring. When a large quantity of reducer is added, a longer time is required to effect complete mixing. Also, separation of the material may occur. Use only reducers recommended by the manufacturer of the finishing material. Incorrect reducer may ruin the material.

5. Mixing of reduced material. Reduced material which has increased in viscosity due to loss of reducer will require further additions of reducer. Reduced pigmented material should be kept thoroughly stirred while in use. Otherwise,

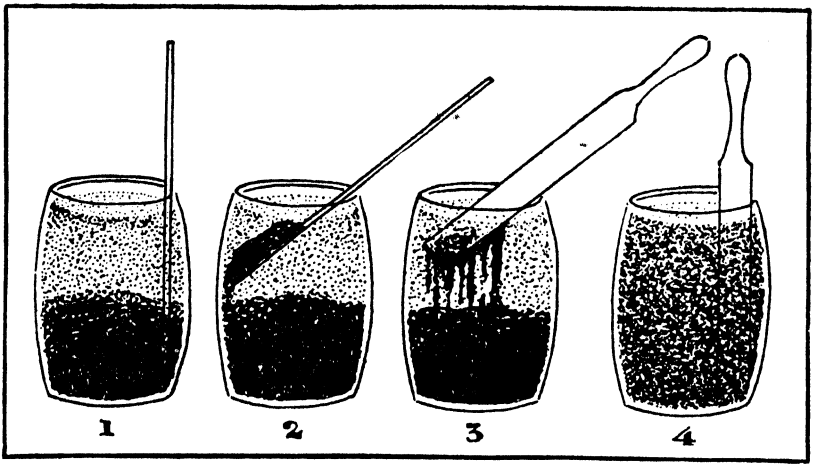


Fig. 2. Mixing in open head drums.

1. Push paddle to bottom, keeping flat side against side of drum.
2. Pull handle backward, lifting material nearly to surface.
3. Twist paddle to right, throwing off material.
4. Bring paddle back edgewise to position 1.

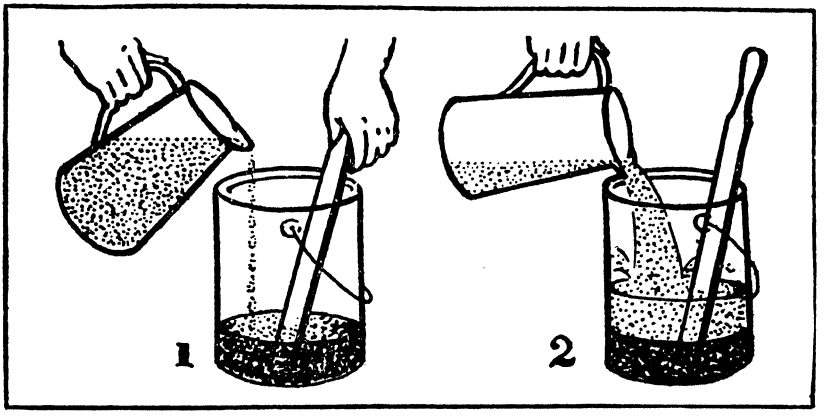


Fig. 3. Reducing finishing materials.

1. Correct Method. Add reducer gradually and with constant stirring.
2. Wrong Method. Add reducer in large quantity without stirring.

pigment settling will produce variations in color, gloss, hiding power and film and application characteristics.

6. *Material in dip tanks.* Reduced material in dip tanks should be stirred frequently while the tank is in use. When not in use, dip tanks should be kept covered, preferably with covers close to the surface.

7. *Test for complete mixing.* Using a paddle or rod long enough to reach the bottom of the container, flow a few drops of material from the top and bottom of the container on a piece of clean glass. Incomplete mixing will be indicated by difference in color and difference in the flow down the glass.

TINTING ALUMINUM FINISHING MATERIALS

Aluminum finishing materials may be tinted with other pigments to obtain different metallic color effects. One of the purposes of tinting is to secure a good contrast between succeeding coats of aluminum finishing material in order that the second coat will be uniformly applied. Tinted aluminum finishing materials may also be employed for their decorative effect. Practically any colored pigment may be used for this purpose though some give more color change than others, depending on their tinting strength. To obtain strong tints, the proportion of aluminum pigment may be reduced. Allowing the mixed tinted finishing material to stand 24 to 48 hours will intensify the color and produce more uniform and consistent results.

The tinting pigments which have been found to give the best results are as follows:

Prussian Blue produces a deep blue tint (recommended for best color contrast).

Chrome Green produces a green tint.
Chrome Yellow produces a light green tint.
Yellow Ocher produces a light yellow tint.
Carbon Black produces a gun-metal gray.
Lead Carbonate produces a battleship gray.
Spanish Iron Oxide produces a light pink tint.
Toluidine Red produces a rose tint.

The tinting pigments should be added to the vehicle and thoroughly incorporated before the vehicle is mixed with the aluminum. A few experiments will give the exact quantities necessary to obtain the desired effect. The durability of these tinted aluminum finishing materials compares very favorably with untinted aluminum finishing materials, provided the amount of aluminum pigment is not reduced more than 20 per cent. The permanency of color of these tinted aluminum finishing materials depends entirely on the resistance to fading of the tinting pigments.

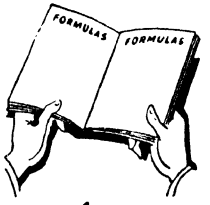
(Courtesy Aluminum Company of America.)

MIXING ALUMINUM FINISHING MATERIALS

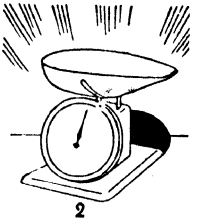
1. Check the recommended formula for the proper proportions of vehicle and aluminum powder or paste. Carefully estimate the quantity of finishing material that will be required for the day's work. If, for unavoidable reasons, the quantity prepared is not used entirely, the remainder may be mixed with fresh material in amounts up to ten per cent.

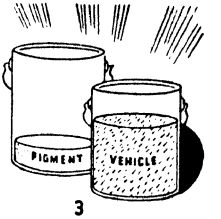
2. Carefully weigh out the necessary amount of powder or paste. Do not measure by volume or guess at the weight.

3. Place the weighed powder or paste in the bottom of a clean container large enough to accommodate the whole mix and allow room for stirring. In a

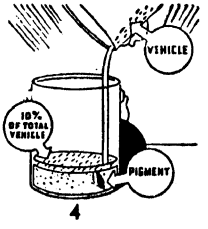


1

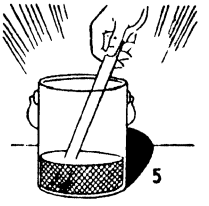

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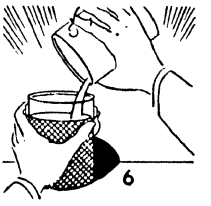


3



4


5


6


7

separate clean container measure out the exact volume of vehicle to go with the powder or paste.

4. Pour the vehicle over the powder or paste in small quantities. Never add the powder or paste to the vehicle. In mixing the vehicle and powder or paste it is important that at first the vehicle be added in small amounts and that each quantity be thoroughly stirred before more vehicle is added. Use about ten per cent of the vehicle for the first addition, ten per cent for the second addition, twenty per cent for the next, and finally the remaining sixty per cent.

5. Stir the completed combination thoroughly.

6. Box by pouring from the mixing container into the vehicle container and back several times.

7. As a final operation, strain through double- or triple-thickness cheesecloth to be certain that no unbroken lumps remain.

During application the aluminum finishing material should be stirred but this stirring should be moderate and no more than is necessary to keep the metallic pigment in suspension. Too much agitation may tend to cause darkening and loss of leafing power.

(Illustrations courtesy Aluminum Company of America.)

CONVERSION TABLE

DEGREES BAUME (AMERICAN)—SPECIFIC GRAVITY (at 60°F.)

For finishing materials lighter than water. Degrees Baumè $= \frac{140}{\text{Sp. Gr.}} - 130$

°Be	0	1	2	3	4	5	6	7	8	9
10	1.000	0.993	0.986	0.979	0.972	0.966	0.959	0.952	0.946	0.940
20	0.933	0.927	0.921	0.915	0.909	0.903	0.897	0.892	0.886	0.880
30	0.875	0.870	0.864	0.859	0.854	0.848	0.843	0.838	0.833	0.826
40	0.824	0.819	0.814	0.809	0.804	0.800	0.795	0.791	0.786	0.782
50	0.778	0.773	0.769	0.765	0.761	0.757	0.753	0.749	0.745	0.741
60	0.737	0.733	0.729	0.725	0.722	0.718	0.714	0.711	0.707	0.704
70	0.700	0.696	0.693	0.690	0.686	0.683	0.680	0.676	0.673	0.670
80	0.667	0.664	0.660	0.657	0.654	0.651	0.648	0.645	0.642	0.639
90	0.636	0.633	0.631	0.628	0.625	0.622	0.619	0.617	0.614	0.611

For finishing materials heavier than water. Degrees Baumè $= 145 - \frac{145}{\text{Sp. Gr.}}$

°Be	0	1	2	3	4	5	6	7	8	9
0	1.000	1.007	1.014	1.021	1.028	1.036	1.043	1.051	1.058	1.066
10	1.074	1.082	1.090	1.098	1.107	1.115	1.124	1.133	1.142	1.151
20	1.160	1.169	1.179	1.188	1.198	1.208	1.218	1.229	1.239	1.250
30	1.261	1.272	1.283	1.295	1.306	1.318	1.330	1.343	1.355	1.368
40	1.381	1.394	1.408	1.422	1.436	1.450	1.465	1.480	1.495	1.510
50	1.526	1.543	1.559	1.576	1.593	1.611	1.629	1.648	1.667	1.686
60	1.706	1.726	1.747	1.768	1.790	1.812	1.835	1.859	1.883	1.908

STORING AND HANDLING FINISHING MATERIALS

1. Use Old Shipments of Finishing Materials First

Finishing materials may deteriorate on long storage. Date all containers of finishing materials on delivery and do not draw from a new shipment until all previous lots have been used.

2. Store Finishing Materials Properly

Extremes of heat or cold may harm finishing materials. Maintain a temperature of at least 60°F. in paint storage rooms but do not expose materials to the direct heat of steam radiators, etc. Do not store finishing materials outside. Allow all finishing materials to come to room temperature before using.

3. Mix Finishing Materials Thoroughly Before Thinning

Incomplete mixing is often the cause of finish troubles such as streaky color, poor adhesion, variable gloss, poor hiding, etc. Mix all finishing materials thoroughly before adding thinners, especially pigmented materials in which settling may have occurred.

4. Thin Finishing Materials Properly

Many finishing difficulties arise from the use of improper thinners or from adding thinners incorrectly. In all cases use the thinner recommended by the

manufacturer of the finishing material. Add thinners slowly and in small amounts, making certain that each addition is stirred in thoroughly.

5. Do Not Waste Finishing Materials or Thinners

To waste finishing materials or thinners is to waste valuable resins, pigments, solvents, etc. Prepare only enough material for one day's use. Do not spill finishing materials. Cover all containers tightly when not in use to prevent skinning and evaporation of volatile solvents. Completely empty all containers.

6. Maintain Good Housekeeping

A dirty shop is neither pleasant, efficient nor safe. Clean up all spilled material. Keep equipment clean and in good operating order. Employ scrap cans. Keep work arranged in orderly fashion.

CARE AND MAINTENANCE OF SPRAYING EQUIPMENT

Spray guns and spraying equipment are precision made tools. Handle and care for them accordingly.

Clean spray gun at end of each day's use.

Do not immerse spray gun completely in thinner. To do so dries out packings and removes lubrication.

Caustic paint removers attack metals and should not be used.

Clean air cap by immersing in thinner. Use a soft wood stick or broom straw if holes become slogged. Do not injure gun by using metal pins or wire.

Unnecessary roughness or careless dropping may cause spray gun to become permanently misaligned. Handle with care, hanging the gun up on a proper hook when not spraying.

Lubricate gun regularly as recommended by the manufacturer.

Have on hand parts list and replacements. Return gun to manufacturer for all major repairs.

Do not dismantle gun (except for cleaning air cap) unless absolutely necessary.

Coil hoses up when not in use.

Avoid dragging hose through oil, grease and dirt.

Don't use hoses as rope to pull equipment around.

Don't use material hose as an air hose or vice versa.

Release tension on transformer mechanism when idle by turning out adjusting screw or adjusting knob.

Drain moisture from transformer daily. Replace filter packs regularly.

Keep transformer clean.

Drain air receiver of compressor daily.

Remove paint and dirt accumulation around compressor which reduces efficiency and may do harm to belts and electrical equipment.

Place compressor where it gets fresh air and keep air strainers clean.

Keep the interior of pressure feed tanks and cups and suction cups clean. Dried paint particles and skins left in tank may cause clogging of spray gun.

Release tension on regulator of pressure feed tank by turning adjusting screw out when not in use.

Damaged or leaky valves in pressure feed tanks and cups should be replaced to eliminate loss of material or air and do away with the hazard involved.

Keep the vent open at all times (suction feed).

Avoid hard knocks which cause denting and damaging of cups and tanks.

Keep spray booths clean. Paint accumulations do not contribute to good finishes and present fire hazards.

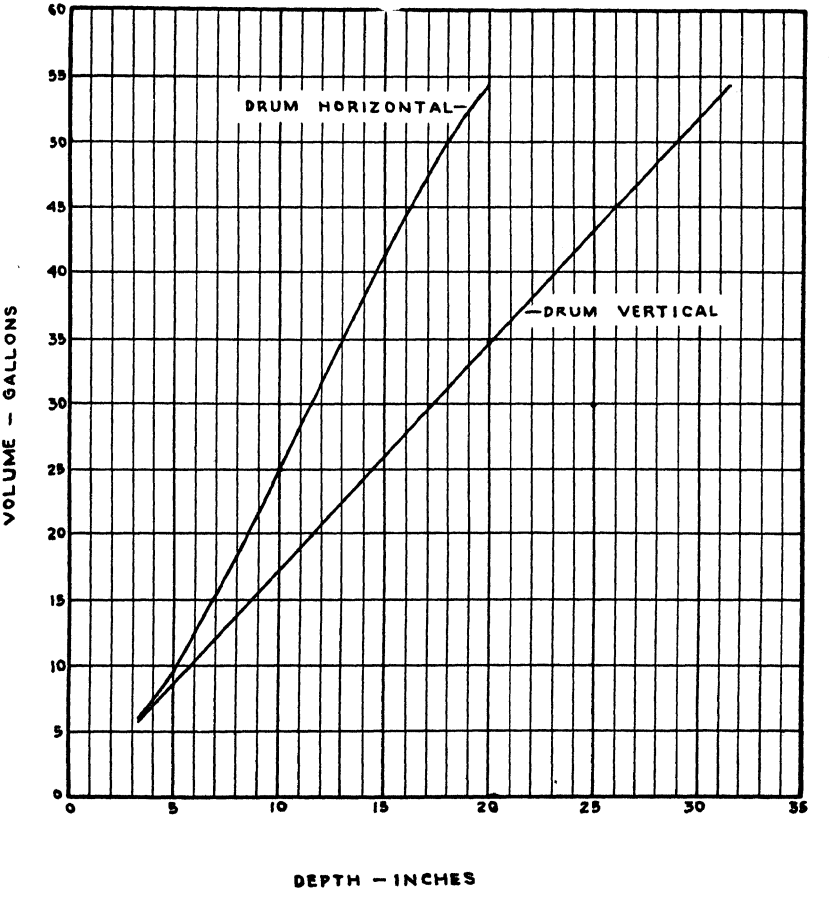
Keep spray booths free of paint pails, jigs, etc. which interfere with smooth movement of work in and out of booth.

Clean spray booth fan and lubricate bearings regularly (unless bearings are grease sealed type).

DRUM VOLUME CHART

The following drum volume chart will be found useful for quickly and easily determining the approximate contents of 55 gallon (34⅞" x 24") drums containing finishing materials, thinners, etc.

Any suitable measuring stick marked in inches, such as an ordinary yardstick, may be used. The measuring stick is inserted into the drum through the top opening (if the drum is in the vertical position) or through the side opening (if the drum is horizontal) with the one inch graduation down. Care must be taken that the stick is straight, that it is held vertically and that it touches the bottom of the drum before being withdrawn. On withdrawal, the depth in inches on the stick is noted and reference to the chart will show the approximate number of gallons of liquid in the drum.

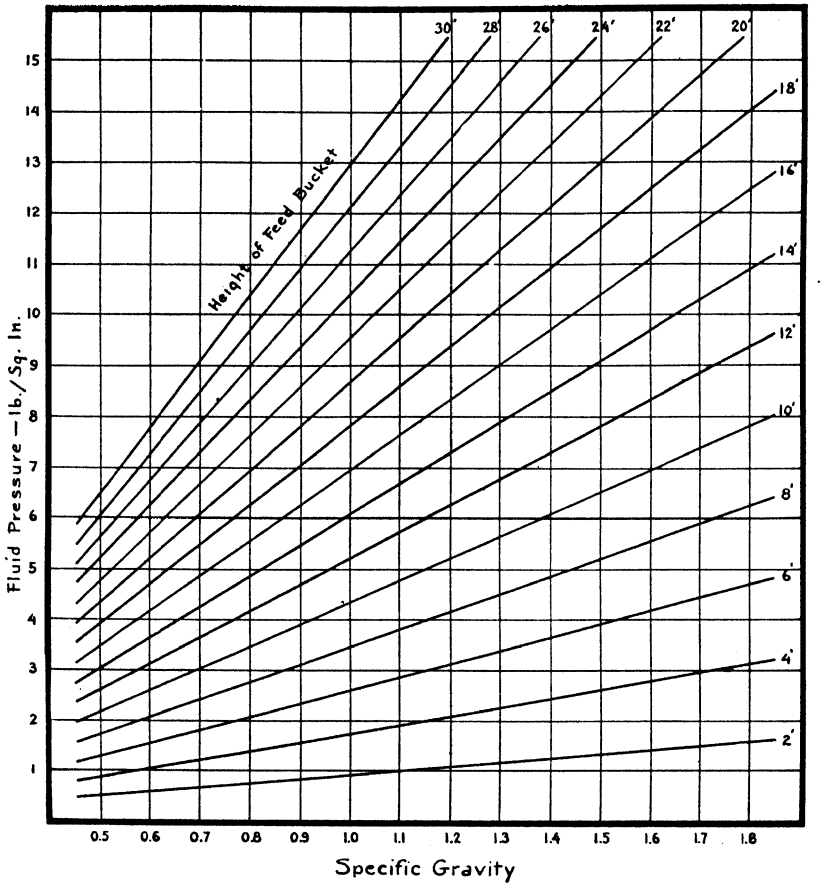


SPRAY GUN FLUID PRESSURE CONVERSION CHART

The following fluid pressure to gravity bucket height conversion chart will be found useful in determining the height to which a gravity material feed bucket must be raised to approximate a given fluid pressure.

To make the conversion using the chart, a horizontal line is drawn from the fluid pressure scale at the pressure it is desired to approximate. A vertical line is drawn from the specific gravity scale at the value of the specific gravity of the material to be sprayed. The intersection of these two lines will fall on or near one of the sloping lines which give the approximate height (from the surface of the material in the bucket to the spray gun nozzle).

For example, to approximate a fluid pressure of 5 pounds per square inch with a finishing material having a specific gravity of 0.82, it is necessary to raise the bucket to a height of approximately 14 feet.



PRESSURE LOSS IN SPRAY GUN AIR HOSES

Air pressure loss in spray gun air hoses is the difference between the air pressure at the gun and at the point where the air hose connects with the air line. The following table shows air pressures at the gun (assuming 12 cubic feet per minute consumption at 60 pounds per square inch) compared with air pressures at the point of connection with the air line for various lengths of two common sizes of hose.

Line Pressure (lb./sq. in.)	Pressure at Gun (lb./sq. in.)					
	5′	10′	15′	20′	25′	50′
40	34.00	32.00	30.50	29.00	27.75	16.00
50	42.50	40.00	38.00	36.00	34.00	22.00
60	51.00	47.50	45.50	43.25	41.00	29.00
70	59.25	55.50	53.00	50.50	47.50	36.00
80	67.75	63.50	60.50	57.50	54.50	43.00
90	76.00	71.25	68.00	64.75	61.00	60.50
40	37.75	37.25	36.75	36.50	36.00	31.50
50	47.00	46.50	46.00	45.50	45.00	40.00
60	56.25	55.50	55.00	54.50	54.00	48.50
70	65.50	64.75	64.00	63.25	62.75	57.00
80	74.00	73.75	73.00	72.00	71.25	65.50
90	83.00	82.50	81.50	80.50	79.50	74.00

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SPRAY FINISHING PROBLEMS

Causes and Corrections

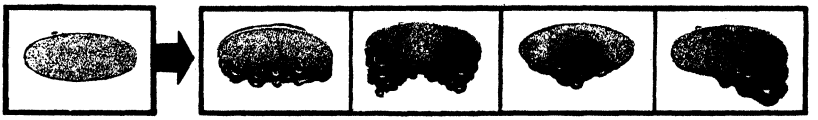
The successful application of sprayed coatings depends on handling technique and proper adjustment and maintenance of spraying equipment. Various spraying troubles and their causes are given below.

1. Fluid leakage from needle packing nut.
 a. Loose nut.
2. Fluid leakage from front of gun.
 a. Dry packing.
 b. Needle not seating.
 c. Worn tip.
 d. Dirt on tip.
 e. Broken needle spring.
 f. Packing nut too tight.
3. Air leakage.
 a. Air valve not seating.
 b. Dirt on valve or seat.
 c. Worn valve or seat.
 d. Broken spring.
 e. Sticking valve stem.
 f. Tight packing nut.
4. Jerky or fluttering spray.
 a. Air leakage into fluid line.
 b. Insufficient material in container.
 c. Container tipped at acute angle.
5. Heavy top pattern.
 a. Horn holes partially plugged.
 b. Obstruction on top of fluid tip.
 c. Dirt or dry paint on air cap seat or fluid cap seat.
6. Heavy bottom pattern.
 a. Horn holes partially plugged.
 b. Obstruction on bottom side of fluid tip.
 c. Dirt or dry paint on air cap seat or fluid cap seat.
7. Heavy right side pattern.
 a. Right side of horn holes partially plugged.
 b. Dirt on right side of fluid tip.
 c. Right jet clogged (twin jet cap).
8. Heavy left side pattern.
 a. Left side of horn holes plugged.
 b. Dirt or dry paint on left side of fluid tip.
 c. Left jet clogged (twin jet cap).
9. Heavy center pattern.
 a. Low setting of spreader adjustment valve.
 b. Low atomizing pressure or high material viscosity (twin jet cap).
10. Split spray pattern.
 a. Air and fluid improperly balanced. Reduce width of spray pattern or

increase fluid pressure. Increasing fluid pressure requires increased speed of handling.

11. Orange peel finish.
 a. A common cause is improper thinner. A slow evaporating thinner usually eliminates this fault.
12. Streaks in finish.
 a. Tipping gun. One side of pattern hits surface from shorter distance causing more material to be applied at this point.
 b. Air cap or fluid tip may be dirty or burred causing heavy top or bottom pattern.
 c. Split spray causing heaviness at top and bottom of pattern. Reduce width of spray or increase fluid pressure.
13. Runs and sags.
 a. Too much material. Reduce fluid pressure or increase operating speed.
 b. Gun tilted at angle. More material is supplied where pattern is closest to surface.
 c. Excessive thinning of material or too slow evaporating thinner.
14. Mist or fog.
 a. Over atomization due to:
 1. Too high air pressure.
 2. Wrong air cap for material being sprayed.
 3. Wrong fluid tip for material being sprayed.
 4. Fluid pressure too low (pressure feed).
 b. Improper use of gun due to:
 1. Incorrect stroking.
 2. Gun too far from surface.
15. Other finishing troubles due to:
 a. Insufficient atomization.
 b. Material not thoroughly dissolved or agitated.
 c. Drafts in finishing room.
 d. High humidity.

TESTING SPRAY PATTERNS



SPRAY THIS PATTERN FOR 2 TO 5 SECONDS —THEN OBSERVE THE SAG THAT FORMS.

CORRECT PATTERN: —PARTICLES EVENLY DISTRIBUTED. —NO LARGE PARTICLES ON FRINGE OF PATTERN.

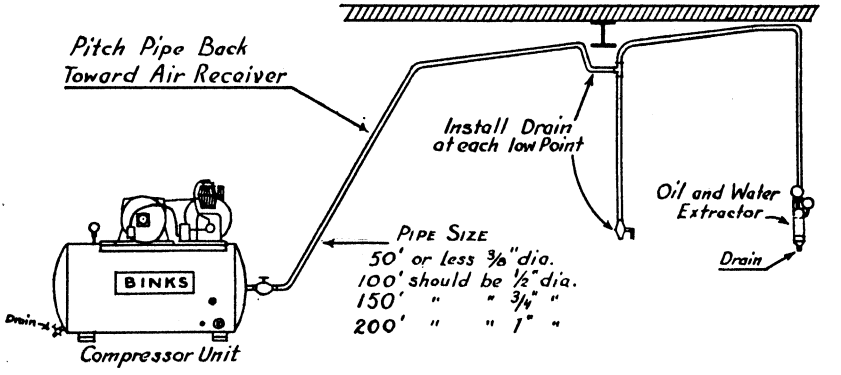
SPLIT SPRAY: —TOO MUCH AIR TO OUTSIDE PORTS. —MATERIAL TOO THIN FOR AIR CAP, OR TOO HIGH AIR PRESSURE

HEAVY CENTER: —INSUFFICIENT AIR TO OUTSIDE PORTS —TOO MUCH MATERIAL FLOW. —TOO LOW AIR PRESSURE.

HEAVY END: —CAUSED BY DISTORTED AIR STREAM, BY DIRTY AIR CAP OR BY DIRTY FLUID TIP.

(Courtesy Westinghouse Electric & Mfg. Co.)

COMPRESSED AIR SYSTEM FOR SPRAY GUNS



The above diagram illustrates a proper arrangement for a compressed air system for spray guns. It is poor practice to mount the oil and water extractor on or even near the compressor. The temperature of the air is greatly increased as it passes through the compressor and if the air is still warm when it passes through the extractor the moisture in it will not be removed effectively with the result that, as the air cools in the hose beyond the extractor, it will condense and cause a faulty finish. The extractor should be mounted at least 25 feet from the compressor.

Air lines should be pitched back toward the compressor so that condensed moisture will flow back into the air receiver where it can be removed through a drain cock. Every low point in the air line acts as a water trap and each such point should be fitted with a drain cock.

(Illustration courtesy Binks Manufacturing Co.)

SPRAY OPERATOR EVALUATION

1. TIME UTILIZATION FACTOR

Time utilization factor is the ratio of the amount of finishing material actually sprayed to the amount which could be sprayed in the same time with the gun in continuous operation. It is expressed by the equation:

Eq. 1

$$Tu = \frac{Wa}{Wp}$$

Tu—time utilization factor
Wa—finishing material actually sprayed per hour - pounds
Wp—finishing material possible per hour - pounds

Example

An operator sprays 6.0 pounds of lacquer per hour (Wa). At the adjustment used, the gun in continuous operation could deliver 10.0 pounds of lacquer per hour (Wp).

Substituting these data in Equation 1, the time utilization factor is:

$$Tu = \frac{6.0}{10.0} = 0.6$$

That is, the operator is spraying 60% of the time. During 40% of the time the gun is not functioning.

2. MATERIAL UTILIZATION FACTOR

Material utilization factor is the ratio of the amount of finishing material actually applied to the amount sprayed. It is expressed by the following equation:

Eq. 2

$$Mu = \frac{(W)\ (N)}{(Wa)\ (Ps)}(100)$$

Mu—material utilization factor
W—cured finishing material on average piece - pounds
N—number of pieces finished per hour
Wa—(as above)
Ps—per cent by weight of non-volatile solids in finishing material
100—constant

Example

A spray operator finishes 450 pieces per hour (N), using 6.0 pounds of lacquer (Wa). The lacquer contains 25.0% by weight of non-volatile solids (Ps). After drying, the lacquer on the average piece was found to weigh 0.0012 pounds (W). (*Note.* Find (W) by weighing piece before finishing and after finish is applied and cured.)

Substituting these data in Equation 2, the material utilization factor is:

$$Mu = \frac{(0.0012)\ (450)}{(6.0)\ (25.0)}(100) = 0.36$$

That is, of the material sprayed, 36% is useful while the remainder is lost in overspray.

3. PRODUCTIVITY FACTOR

Productivity factor is the ratio of the number of pieces actually finished to the number which could be finished in the same time if the gun were in continuous operation and there were no overspray losses, etc. It is the product of the time

utilization factor and the material utilization factor and is expressed by the following equations:

Eq. 3

$$P = (Tu)\ (Mu)$$

Eq. 4

$$\text{or, } P = \frac{(W)\ (N)}{(Wp)\ (Ps)}(100)$$

P—productivity factor
(other factors as above)

Example

Taking data from the above examples and substituting in Equations 3 and 4, the productivity factor is:

$$P = (0.6)\ (0.36) = 0.216$$

$$\text{or, } P = \frac{(0.0012)\ (450)}{(10.0)\ (25.0)}(100) = 0.216$$

That is, the operator is producing 21.6% of the number of pieces theoretically possible.

(*Note.* In Equation 4 the quantity (Wa) does not appear and it is not necessary to make measurements of the amount of finishing material sprayed by the operator.)

PREVENTING LACQUER BLUSH

It is possible to select solvents and combine them in a lacquer to give excellent resistance to blushing even under the worst conditions of temperature and humidity. However, there may be distinct disadvantages in using such materials. The cost may be too high. More important, there may be a sacrifice of working and drying properties of the lacquer. It is usually better, therefore, to approach the problem of moisture blush through control in the finishing room.

The ideal procedure for moisture blush control is the complete air conditioning of the room where lacquers are applied. This, of course, involves an expense which is not usually warranted since blushing occurs only during a few months of the year, usually the warm and humid summer months. If air conditioning is not feasible, there are protective measures which can be taken. Given below are a number of suggestions for preventing and remedying blushing.

1. Warm the room in order to reduce the relative humidity of the air.
2. Eliminate all draughts in the vicinity of the work by closing doors and windows and, if necessary, by hanging draught curtains. Movement of air across the wet film will cause faster evaporation of the solvents and have a greater cooling effect.
3. Warm the work either before or after application of the lacquer. If the work is warmed in an oven after finishing, turn off the blower. In this connection it will be found that large heavy work will not blush as easily as light small pieces. The larger pieces do not lose heat as rapidly as do small pieces and consequently cooling is not as rapid.
4. Use a lacquer retarder—a solvent of such properties that some remains in the film until after the temperature has risen above the dew point and any moisture

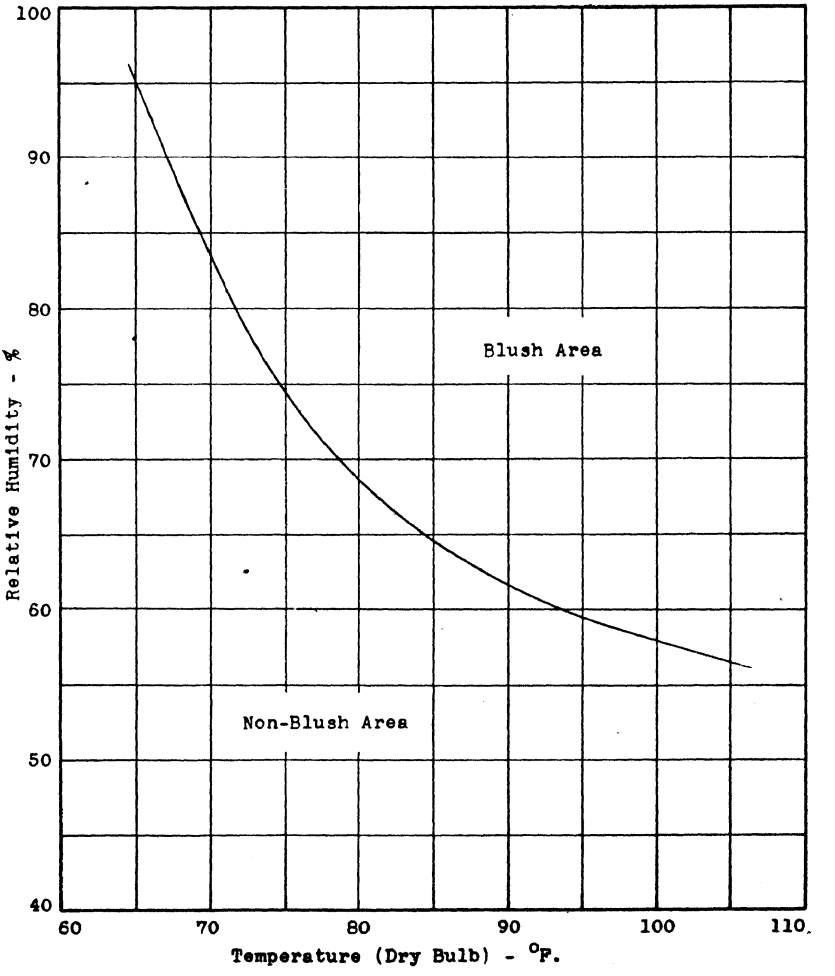
which has condensed on the film has evaporated. Depending on the severity of conditions, lacquer retarder is used in concentrations of from five to thirty per cent by volume of the thinner lacquer.

5. Bring all lacquer to room temperature before beginning application. Keep all containers tightly closed.

6. Regularly drain all condensed moisture from air supply lines leading to spray guns.

BLUSHING CHART

The following chart shows a typical blush curve for a representative lacquer



of good blush resisting characteristics. Any condition of temperature and relative humidity within the area above the curve will produce blushing. Blushing will not occur under conditions below the curve.

Several temperature and relative humidity readings daily over a period of time, along with notations of the current behavior of the film, will provide data which will permit plotting an exact curve of blush probability for a lacquer applied in a particular finishing department.

RELATIVE HUMIDITY TABLE

Difference — °F.

(Dry Bulb Temp. — Wet Bulb Temp.)

Dry Bulb Temp.—°F.	0°	2°	4°	6°	8°	10°	12°	14°	16°	18°	20°
60	100	89	78	68	58	48	39	30	21	13	5
62	100	89	79	69	59	50	41	32	24	16	8
64	100	89	79	70	60	51	43	35	26	19	11
66	100	90	80	71	61	53	44	37	29	21	15
68	100	90	81	71	63	54	46	38	31	24	16
70	100	90	81	72	64	56	47	40	33	25	19
72	100	91	82	73	65	57	49	42	35	28	21
74	100	91	82	74	66	58	51	44	37	29	23
76	100	91	83	74	67	59	52	45	38	31	25
78	100	91	83	75	67	60	53	46	40	33	27
80	100	91	83	75	68	61	54	47	41	35	29
82	100	92	84	76	69	62	55	49	43	36	30
84	100	92	84	77	70	63	56	50	44	37	32
86	100	92	85	77	70	63	57	51	45	39	33
88	100	92	85	77	71	65	58	52	47	41	35
90	100	92	85	77	71	65	59	53	47	42	36
92	100	92	85	78	72	65	59	54	49	44	39
94	100	93	85	79	72	66	60	54	50	45	40
96	100	93	86	79	73	67	61	55	50	46	41
98	100	93	86	79	73	67	61	56	50	46	42
100	100	93	86	80	73	68	62	56	51	46	42

NUMERICAL RATING OF ORGANIC FINISH QUALITY

The following is a method for comparing the quality of two or more organic finishes on a numerical basis. The method is used for the selection of the best available finish for any particular application.

Step 1. Select those properties which are necessary for satisfactory field performance of the finish. For example, a piece of equipment may require a finish having alkali resistance, hardness, humidity resistance, gloss retention, discoloration resistance and oil resistance.

Step 2. Assign to each property selected, a percentage value relative to the other properties which will insure proper field performance of the finish as a

whole. For example, the properties chosen above may be given the following values.

PROPERTY	PERCENTAGE RELATIVE VALUE
Alkali resistance	35
Hardness	25
Humidity resistance	15
Gloss retention	10
Discoloration resistance	10
Oil resistance	5
	100%

Step 3. Test the finish under consideration for each property and evaluate each property on the following scale:

	TEST RATING
Very poor	0
Poor	2
Fair	4
Good	6
Very good	8
Excellent	10

Step 4. Multiply the percentage relative value of each property by its test rating. This gives the final value in "points" for each property. The sum of such points of all of the properties is the final numerical value of the finish. The best possible score would, of course, be 1000 points if each property selected obtained a rating of excellent in test.

A typical set of data is given below:

PROPERTY	PERCENTAGE RELATIVE VALUE		TEST RATING		POINTS
Alkali resistance	35	×	8	=	280
Hardness	25	×	4	=	100
Humidity resistance	15	×	6	=	90
Gloss retention	10	×	6	=	60
Discoloration resistance	10	×	2	=	20
Oil resistance	5	×	8	=	40
					Total 590

Final Numerical Value: 590 points.

LIGHT REFLECTION VALUES OF PAINT COLORS

The following light reflection values of paint colors, selected for their adaptability to industrial use, were obtained on a photometer in which the light falls on the sample normally and is reflected at forty-five degrees. This method measures essentially the diffused reflection from a surface as distinguished from glare or mirrorlike reflection.

White	84.0
Cream	68.8
Ivory	66.7
Light Pink	66.5
Yellow	57.0
Flesh	51.6
Buff	51.5
Light Gray	51.5
Light Green	45.2
Aluminum Gray	41.0
Light Blue	36.4
Sage Green	36.3
Brown	27.5
Dark Red	13.8
Dark Green	9.2
Dark Blue	8.4

(Courtesy The New Jersey Zinc Company.)

LUMINESCENT FINISHING MATERIALS

Terms and Definitions

The expanding use of luminescent finishing materials in business, industrial and home applications has brought to the finishing industry a new nomenclature which must be known and understood for efficient use and handling of these materials. Given below are the important terms and definitions.

1. **Light Source.** Any object, surface or substance which emits light is known as a light source. An electric light bulb in use, for example, is a light source. The sun, of course, is the most widely used light source. Luminescent finishing materials, therefore, are light sources since they emit light.

2. **Incandescence.** The emission of light as the result of intense heat. A tungsten filament lamp bulb, such as is used in most electric lighting systems, is an example of incandescent light.

3. **Luminescence.** The emission of light not due to incandescence. A luminescent light is a "cold" light. Luminescent finishing materials, therefore, emit "cold" light.

4. **Fluorescence.** The emission of luminescent light only during the time the luminescent finishing material is exposed to an exciting light source.

5. **Phosphorescence.** The emission of luminescent light continuing for a

time after the exciting light has been extinguished. "Afterglow" is a term often used for phosphorescence.

6. **Wavelength.** Light or radiant energy travels with a wave-like motion. The wavelength is the distance from the crest of one wave to the crest of the next wave.

7. **Ultraviolet Light.** Radiant energy of shorter wave length than visible light.

8. **Near-Ultraviolet Light.** That portion of ultraviolet light nearest the visible portion of the spectrum. It contains some visible light of the shortest wave length.

9. **"Black" Light.** The popular name for near-ultraviolet light, so called because the major portion of the energy is invisible to the eye.

10. **Exciting Light.** Any light that will induce luminescence in responsive materials. Thus, "black" light is an exciting light. It is also called an "activating" light.

11. **Angstrom Unit.** A unit of length used to express the wave length of light. Its symbol is A. U. One Angstrom unit is 0.00000001 centimeter or 0.000000004 inch (approx.). The wave lengths of visible light range from 3900 Angstrom units to 7700 Angstrom units. Ultraviolet wave lengths measure from about 150 to 3900 Angstrom units. Infra-red wave lengths run upwards from 7700 Angstrom units.

12. **Millimicron.** Another unit of length sometimes used in place of the Angstrom. One millimicron equals 10 Angstrom units or one-thousandth of a micron.

13. **Brightness Level.** The term which is used to designate the intensity or brightness of light seen with the eye. The brightness level outdoors in bright sunlight is probably 500,000 times higher than that of full moonlight.

14. **High Brightness Level.** Brightness that is sufficiently high for normal daylight vision.

15. **Low Brightness Level.** Level insufficient for normal vision as, for example, bright starlight.

16. **Footcandle.** Unit used to measure the intensity of light. The brilliance of one footcandle may be visualized by holding a piece of white paper one foot away from the light of an ordinary candle or four feet from a 25-watt lamp. The sun on a clear day at noon produces a brightness level of about 10,000 footcandles.

17. **Microlambert.** Another unit for measuring the intensity of light, used principally to express low intensities. One footcandle equals 1076 microlamberts. The brightness of luminescent finishing materials is usually measured in microlamberts.



FINISHING MATERIAL COSTS

1. MILEAGE PER GALLON

$$M = \frac{(W)\ (Ps)}{(Dc)\ (Tc)\ (519)}$$

M—mileage per reduced (ready to apply) gallon of finishing material—square feet

W—weight per gallon of reduced finishing material—pounds
This datum is most easily obtained with an hydrometer. The specific gravity reading multiplied by 8.33 gives the weight per gallon in pounds.

Ps—per cent by weight of non-volatile solids in reduced finishing material
Carefully weigh a small sample of the reduced finishing material into a tared weighing dish and dry to constant weight at about 100°F. The weight of the dried sample divided by the weight of the original sample and multiplied by 100 gives the per cent by weight of non-volatile solids.

Dc—density of the cured film of finishing material
Accurately weigh and measure the area and thickness of a clean, flat panel. Apply a uniform coat of the finishing material to one side of the panel and, in the case of a baking material, bake for the regular schedule, or, in the case of an air drying material, dry to constant weight. Accurately weigh and measure the thickness of the finished panel.
The density of the cured film from the following equation:

$$Dc = \frac{(w)}{(a)\ (t)\ (16.39)}$$

w—weight of cured film (by difference)—grams
a—area of panel—square inches
t—thickness of cured film (by difference)—inches
16.39—constant

Tc—thickness of cured film of finishing material which is acceptable for protection and/or decoration—inches

519—constant

2. COST PER GALLON

$$C = \frac{(Pt)\ (Ct) + (100\text{-}Pt)\ (Cp)}{100}$$

C—cost per reduced gallon of finishing material—dollars
Pt—per cent by volume of added reducer in reduced finishing material
Ct—cost per gallon of reducer—dollars
Cp—cost per gallon of unreduced finishing material—dollars

Example

An organic finishing material costing $2.00 per gallon (Cp) required a 20.00 per cent by volume reduction (Pt) with a reducer costing $0.85 per gallon (Ct). In this reduced form the material weighed 6.5 pounds per gallon (W) and con-

tained 35.0 per cent by weight of non-volatile solids (Ps). The cured film thickness acceptable for protection and decoration was 0.0013 inches (Tc).

A uniform coat of the reduced finishing material was applied to a clean, flat panel measuring 48 square inches in area (a). The coated panel was then cured. The bare panel measured 0.0469 inches in thickness and weighed 230.15 grams. The finished panel measured 0.0521 inches in thickness and weighed 234.33 grams.

Substituting the above data in the given equations:

$$Dc = \frac{(234.33\text{-}230.15)}{(48)\ (0.0521\text{-}0.0469)\ (16.39)} = 1.025$$

$$M = \frac{(6.5)\ (35.0)}{(1.025)\ (0.0013)\ (519)} = 329 \text{ sq. ft./gal.}$$

$$C = \frac{(20.0)\ (0.85) + (100\text{-}20.0)\ (2.00)}{100} = \$1.77/\text{gal.}$$

Whence, the material cost per square foot finished =

$$\frac{C}{M} = \frac{1.77}{329} = \$0.00538$$

The final calculated material cost per square foot finished, as obtained above, is an absolute figure and does not account for overspray losses, uneven films, etc. It is based on 100% utilization, that is, no material waste and the application of an absolutely uniform film. However, the relative costs of two finishing materials may be obtained from a comparison of the calculated costs. For example, if the calculated cost of material A is $0.0080 per square foot finished and that of material B is $0.0040, then material A is twice as expensive as material B.

Furthermore, if the actual cost and the calculated cost of a material are both known, then the actual cost of any other material whose cost has been calculated is indicated by direct ratio. For example, if the actual cost of material A above is $0.0120 per square foot finished, then the actual cost of material B will be $0.0060 per square foot finished. In this manner the actual cost for any finishing material may be obtained without the necessity for conducting time-consuming production tests.

THICKNESS OF WET FILMS

In some instances the thickness of wet films cannot be measured conveniently. Wet film thickness can, however, be calculated from the following equation:

$$Tw = TC \frac{(833)\ (Dc)}{(Ps)\ (W)}$$

Tw—thickness of wet film—inches
Tc —thickness of cured film—inches
833—constant
Dc —density of cured film
Ps —per cent by weight of non-volatile solids in finishing material
W —weight per gallon of finishing materials—pounds

Example

A finishing material weighing 10.0 pounds per gallon (W) contains 60.0 per cent by weight of non-volatile solids (Ps). After application and curing, the film is found to measure 0.00150 inches in thickness (Tc) and have a density of 1.80 (Dc).

Substituting these data in the above equation, the thickness of the wet film is:

$$Tw = (0.00150) \frac{(833)\ (1.80)}{(60.0)\ (10.0)} = 0.00375 \text{ in.}$$

WEIGHT PER GALLON AND PER CENT SOLIDS OF REDUCED FINISHING MATERIALS

It is possible to obtain the weight per gallon and per cent solids content of reduced finishing materials, in many cases with good accuracy, without actual analyses by the use of the following equations. It is assumed that there is no gain or loss in volume on the addition of reducer to finishing material.

The weight per gallon of reduced finishing materials is calculated from the following equation:

Eq. 1.

$$Wx = \frac{(100 - X)}{100}(Wu) + \frac{(X)}{100}(Wr)$$

The per cent by weight of non-volatile solids of reduced finishing materials is calculated from the following equation:

Eq. 2.

$$Px = \frac{\frac{(100 - X)}{100}(Wu)\frac{(Pu)}{100}}{(Wx)}(100)$$

Wx—weight per gallon of reduced finishing material at (X) per cent by volume concentration of reducer—pounds
X —per cent by volume of reducer in reduced finishing material
Wu—weight per gallon of unreduced finishing material—pounds

Wr—weight per gallon of reducer—pounds

Px—per cent by weight of solids in reduced finishing material at (X) per cent by volume concentration of reducer

Pu—per cent by weight of solids in unreduced finishing material

Example

An unreduced finishing material weighs 10.0 pounds per gallon (Wu) and contains 60.0 per cent by weight of solids (Pu). Reducer weighing 8.0 pounds per gallon (Wr) is added to the extent of 20.0 per cent by volume of the reduced material, i.e., one to four reduction (X).

Substituting these data in Equations 1 and 2,

$$Wx = \frac{(100 - 20.0)}{100}(10.0) + \frac{20.0}{100}(8.0) = 9.6 \text{ lbs./gal.}$$

$$\text{and, } Px = \frac{\dfrac{(100 - 20.0)}{100}(10.0)\dfrac{(60.0)}{100}}{(9.6)}(100) = 50.0\%$$

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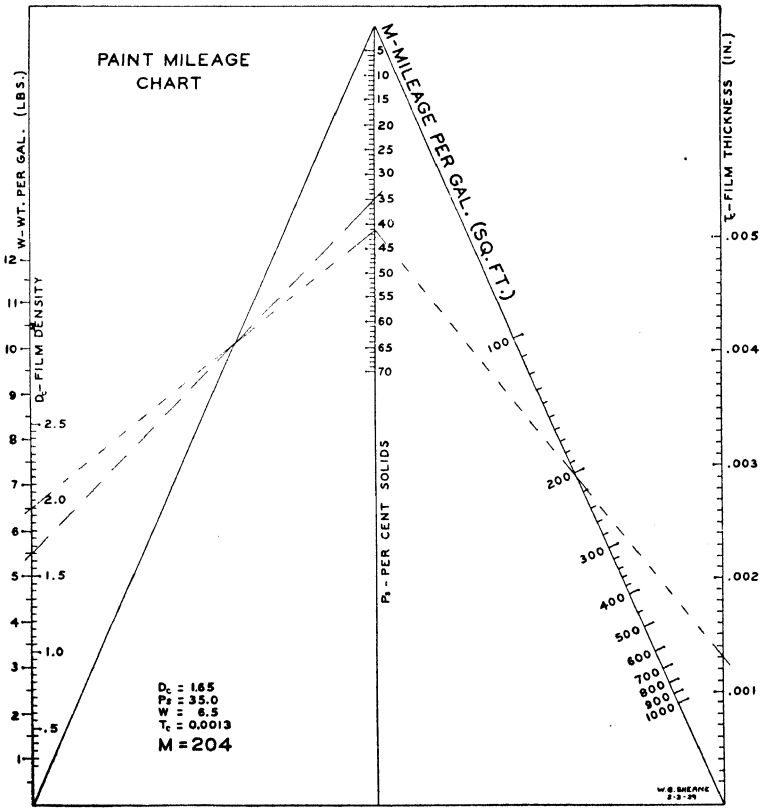
PAINT MILEAGE CHART

Using the data set forth in the lower left hand corner of the chart, for example, the mileage is found as follows:

(1) Draw a straight line connecting the values of Dc (1.65) and Ps (35.0). This line will intersect the sloping line located to the left of the vertical center line.

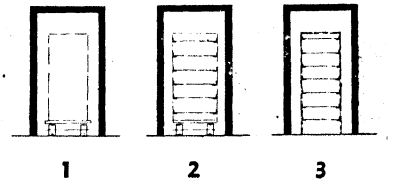
(2) From the value of W (6.5) extend a straight line through the intersection obtained in Step (1) until it intersects the vertical center line.

(3) Draw a straight line connecting the value of Tc (0.0013) and the intersection obtained in Step (2). This line will intersect the mileage scale at the mileage value of M (204 square feet per gallon) for the material under consideration.



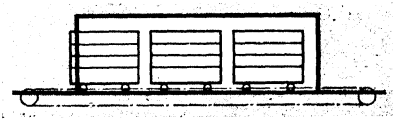
OVENS FOR BAKING FINISHING MATERIALS

The most common types of ovens for baking finishing materials are illustrated and described below. The type to be selected depends on the physical characteristics of the objects to be baked, production requirements, conditions to be maintained in the oven and available building space.



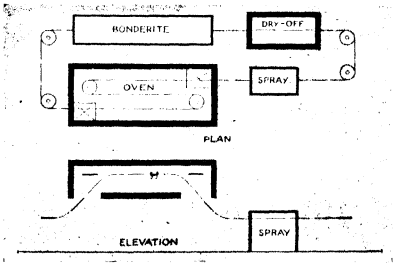
A. Batch Type

1. Trucks
2. Trays on trucks
3. Trays on shelves



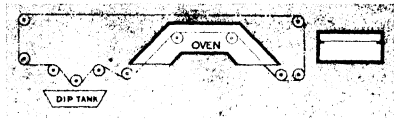
B. Floor Pusher Conveyor Type

1. Continuous
2. Intermittent



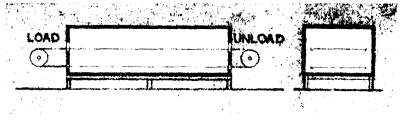
C. Overhead Trolley Type

Pieces may be hung on chain hooks or on trays carried by chain. Multiple passes may be used. May be tied in with other operations. Not limited to horizontal or vertical planes.



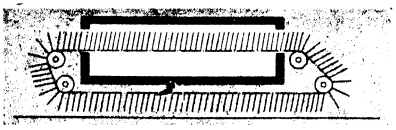
D. Overhead Cross-Bar Conveyor Type

Pieces suspended from cross-bars by hooks. Limited to one plane but may be arranged in vertical or horizontal passes, single or multiple.



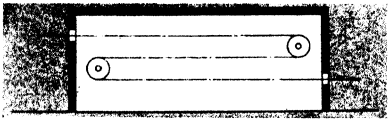
E. Supporting Conveyor Type

Cross-bars, wire mesh screen, solid belt, wires or tapes.



F. Finger Bar Conveyor Type

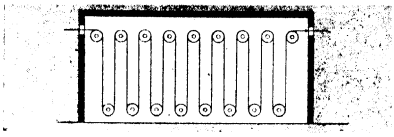
Metal sheets, etc. carried on edge by finger bars supported on chains.



Horizontal Passes

G. Strip Conveyor Type

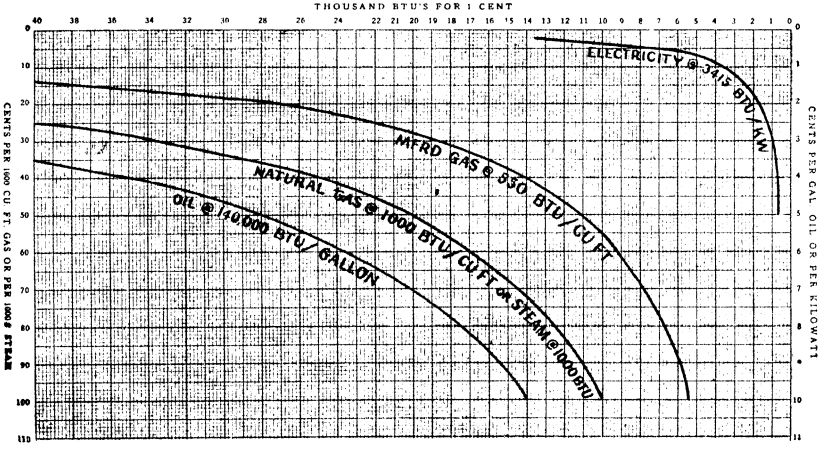
Horizontal or vertical passes, single or multiple. Includes festoon racks, arched pass, series of heated drums, skeleton wheels. (Horizontal and vertical passes illustrated.)



Vertical Passes

(Courtesy J. O. Ross Engineering Co.)

HEAT COST COMPARISON CHART FOR BAKING AND DRYING OPERATIONS



(Courtesy J. O. Ross Engineering Corp.)

Example

For Electricity at 2c per kw, locate line opposite 2c on right-hand side of chart. Follow this horizontal line to left until it intersects "Electricity" curve, then follow vertically to top line at point reading 1.6 or 1,600 BTU's for one cent.

For oil, use same right-hand figures for cents per gallon.

For natural or manufactured gases, use figures at left-hand side of chart for cents per cubic foot and corresponding curves to determine BTU's obtainable for one cent.

FINISHING LIBRARY

The following list of books on organic finishing, finishes and finishing materials has been selected as those most useful to finishers, finishing executives, engineers, chemists, purchasing agents and others who have need for information on finishing materials, methods, metal cleaning and preparation, testing and evaluation, etc.

The list will be changed in succeeding issues of the ***ORGANIC FINISHING HANDBOOK*** as new books are published or the older ones revised. Suggestions for additions to the list will be welcomed. Books may be obtained from the publishers of the ***ORGANIC FINISHING HANDBOOK*** who will send information on prices, etc. on request.

MODERN ORGANIC FINISHES. By Rollin H. Wampler.

The latest book on organic finishing. It is a useful handbook on modern finishing materials, application, drying and baking methods, conveyorization, surface preparation and testing.

SPRAY PAINTING, INDUSTRIAL & COMMERCIAL. By Frederick M. Crewdson.

A practical spraying book useful to both apprentice and experienced finisher. Contains information on conventional as well as specialty finishes and finishing.

SILK SCREEN METHODS OF REPRODUCTION. By Bert Zahn.

Covers the why, what and how of silk screen work—uses and applications, materials, equipment, methods, costs and estimates.

AIRCRAFT ENGINE & METAL FINISHES. By Myron A. Coler.

Thorough and to-the-point descriptions and discussions of metal finishing practices, applicable to all metal finishing operations. A feature is the "Examples" drawn from actual practical experience.

PROTECTIVE COATINGS FOR METALS. By Burns & Schuh.

One of the best books ever published on metal finishing covering the broad subject of technical information on corrosion and corrosion protection.

LACQUER & SYNTHETIC ENAMEL FINISHES. By Ray C. Martin.

Fundamental and practical data on natural and synthetic raw materials and the finishes made from them, along with methods of manufacture, application and testing.

ALUMINUM PAINT & POWDER. By Junius D. Edwards.

The only authoritative book devoted exclusively to aluminum powder and paste and aluminum finishing materials. Written by the outstanding expert on the subject.

PROTECTIVE & DECORATIVE COATINGS. By J. J. Mattiello.

This series provides the best single source of technical information on organic finishing materials, their ingredients, the chemistry of finishes, etc. The various volumes contain all the answers to what finishes are and how they work.

INDUSTRIAL SOLVENTS. By Ibert Mellan.

The most complete book on the physical and chemical characteristics, properties, uses, etc. of solvents used in the finishing industry. Contains an excellent discussion on solution and each chapter has a useful bibliography.

THE NATIONAL PAINT DICTIONARY. By Jeffrey R. Stewart.

A most useful reference work containing definitions of finishing terms, pertinent information on names of chemicals, raw materials, methods of analysis, equipment and apparatus employed in the finishing industry.

PHYSICAL & CHEMICAL EXAMINATION OF PAINTS, VARNISHES, LACQUERS AND COLORS. By H. A. Gardner.

The "bible" of testing finishes and finishing materials and testing equipment, this book describes and illustrates test procedures and instruments for everything from raw materials to finish films.

MUNSELL BOOK OF COLOR. (Abridged edition, 2 vols.)

Contains charts of principal, second- and third-intermediate hues arranged in order of constant hues. These volumes are designed to quickly and positively identify colors. A must for all finishers, chemists and others working with colors.

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